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Synthesis and Characterization of Nano Crystalline $Mg_{0.5}Zn_{0.5}Fe_2O_4$ Prepared by Co-Precipitation Method

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ABSTRACT

$Mg_{0.5}Zn_{0.5}Fe_2O_4$ nano crystalline were successfully prepared by employing the co-precipitation method. From XRD it has been fulfilled that single phase spinel structure has been formed at sintering temperature at 900 °C. The particle sizes are in the range of 30 to 40 nm calculated from XRD and SEM graphs. The $Mg_{0.5}Zn_{0.5}Fe_2O_4$ powders prepared at low temperature exhibit good crystal structure, fine grain size and good magnetic properties. The dielectric constant and dielectric loss of sample have shown good frequency stability and low dielectric losses within the measurement range.

1. Introduction

A nano crystalline material has the crystal size in the order of 1 to 100 nm, in which they appear as single or multi-phase poly crystals structures. In wide-ranging there are two approaches to nanoparticles fabrication that are generally referred to as 'top-down' and 'bottom-up'. The physical methods that comprise techniques like laser ablation or mechanical attrition etc. are cost intensive. Chemical methods have play a main role in rising new materials with novel and technically significant properties; their major advantages are the flexibility of chemical synthesis in design new materials and the fact that chemical processes offer good homogeneity due to mixing at the molecular level [1]. The chemical synthesis method mainly involves sol-gel [2], co-precipitation method [3], and hydrothermal method [4]. These methods are preferably suited for precise control of size and shape of nano phases.

Chemical co-precipitation has extensively worn in manufacturing and research to produce nanometer sized oxide powders. The procedure starts from dissolving salts, which contain metal ions, in a liquid medium. The solution is then mixed with a solution of dissolved precipitating agent, such as oxalic acid or ammonium hydroxide, in order to precipitate the metal oxalates or hydroxides. The final crystalline oxide is then obtained by firing the precipitates at a higher temperature. A noteworthy problem, which has to be to conquer in chemical precipitation, is the agglomeration of the particles in the solution. Thus, dispersants are often added in the reaction process to provide repulsive (electrostatic or steric) interactions between the particles to prevent them from adhering to each other. To eliminate following neck formation and aggregation in the calcinations and firing steps, organic solvents such as ethanol may be added in the final washing step to replace water adsorbed on the powder surface. The advantages of the precipitation method include technical simplicity, low manufacturing cost, high reproducibility and fine particle size. Disadvantages are the difficulty to control the final chemical composition of the products and up scaling issues. In addition, the repeated washing and separation steps make precipitation a time-consuming method [5-9].

Ferrite materials have distinctive electric and magnetic properties, which facilitate them to have a wide range of technical and scientific applications such as magnetic fluids, microwaves components, high-frequency devices, magnetic data storage, biomedical potential applications include drug delivery and as contrast agents in magnetic resonance imaging (MRI) [10]. The most popular type is the cubic spinel structure which has tetrahedral A site and octahedral B site in the AB_2O_4 crystal structure. It is because their dielectrical properties are easy to control as it depends regularly on the variation of the compositions and cation distribution. Moreover, it often has high permeability in the Radio-Frequency (RF) region, high electrical resistivity, chemical stability, mechanical hardness, and reasonable cost [11]. In fact, the application of ferrite as a magnetic feeder to excite an antenna that can cover wide frequency bandwidth from 200MHz-1Hz with different length 0.3-50 m was reported.

Ferrites such as Mg and Zn ferrites are commercially significant materials because of their outstanding electrical and soft magnetic properties. There is a particular application of Mg and Zn ferrites requiring low porosity and controlled microstructure. Ferrites containing Mg and Zn possess higher resistivity value than Mn-Zn one (~107 Ωcm^{-1}) where the resistivity of Mg-Zn ferrite is 106-107 Ω cm^{-1} and make it extra efficient in high frequency. Ferrites are made by sintering a mixture of metal oxides and have the general chemical composition $MO{\cdot}Fe_2O_3$, where M is a divalent metal such as Mn, Mg, Fe, Zn, Ni, and Cd, etc. Relative permeability of several thousand are common [12, 13]. The aim of this present work is to get nanoparticles of $Mg_{0.5}Zn_{0.5}Fe_2O_4$ prepared by co-precipitation method. X-ray diffraction analysis, scanning electron microscopy, TG/DTA and dielectric studies have examined in this study.

2. Experimental Methods

2.1 Chemicals and Apparatus

Magnesium nitrate ($Mg(NO_3)_2$, magnesium nitrate ($Zn(NO_3)_2$, iron nitrate ($Fe(NO_3)_2$, and sodium hydroxide (NaOH) was purchased from sigma Aldrich and used as collected. For confirmation the spinel phase structure, the resulting nano-powders were characterized by means of using XRD with Bruker D8 X-ray diffractometer (CuKα; λ=1.5418 Å). The scanning electron microscopy (SEM JEOL-JSM-7600F) were recorded using (JEM-Model 100CX II) to estimate morphological and crystalline properties of samples. For electrical measurements, the ferrite powders were compressed to pellets of 13 mm diameter and about 5 mm thickness under pressure of 3 tons. The two surfaces of each pellet were coated with silver paint and tested for ohmic contact. The AC electrical conductivity measurement was performed on the pellet samples using Hioki LCR tester 3522-50 (Japan) at room temperatures as a function of frequency (1 KHz–1 MHz).

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2.2 Experimental Procedure for the Synthesis of $Mg_{0.5}Zn_{0.5}Fe_2O_4$

Stoichiometric amounts of magnesium nitrate and ferric nitrate were taken in a 1:2 molar ratio and dissolved in de-ionized water (slowly added) until the solution became neutral. This mixture was co-precipitated at 120 °C for 30 min. The crystalline single phase was formed above 550 °C and the batches were heated in air to 600 °C for 4 h then pressed into a pellet and finally sintered at 1200 °C for 8 h at a heating/cooling rate of 2 °Cmin^{-1} as shown in Fig. 1.

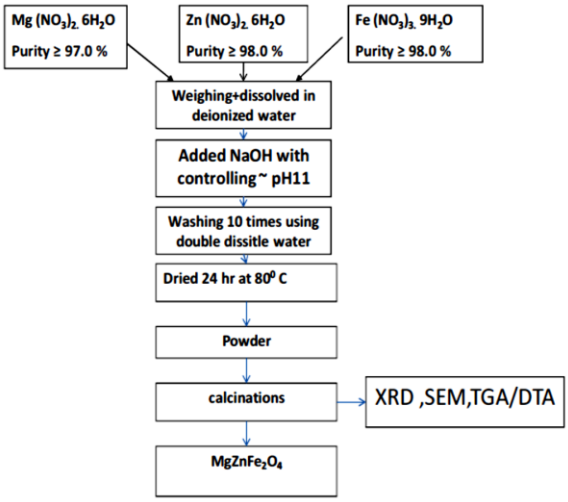

Fig. 1 Flow chart for the co-precipitation synthesis of $Mg_{0.5}Zn_{0.5}Fe_2O_4$

3. Results and Discussion

3.1 X-Ray Diffraction

The XRD pattern of $Mg_{0.5}Zn_{0.5}Fe_2O_4$ nano crystalline are shown in Fig. 2. The presence of peaks in XRD pattern shows that the $Mg_{0.5}Zn_{0.5}Fe_2O_4$ nano and good crystalline in nature. The six peaks in the XRD pattern confirm that the ferrite materials are spinal/cubic in nature. The broadness of the peaks indicates that the size of the material is in the nano range. The crystallite size can be measured from X-ray diffraction patterns by Debye Sheerer equation [14]. The crystalline size varies from 30 nm to 35 nm after calcination and 900 °C sintered respectively. If we analyze XRD patterns, that the (311) peak is the maximum intensity peak shows the single-phase crystal structure of the $Mg_{0.5}Zn_{0.5}Fe_2O_4$ spinel ferrite with a JCPDS-card-04002-5442.

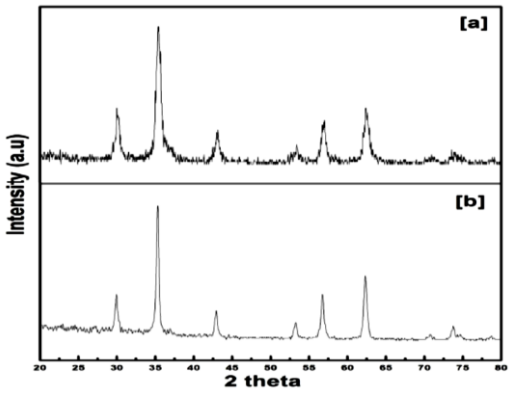

Fig. 2 Schematic diagram of the XRD pattern (a) after calcination (b) after sintering

By increasing temperature of $Mg_{0.5}Zn_{0.5}Fe_2O_4$ form 600 °C to 900 °C, the crystallite size increases and lattice parameter 'a' increases, which corresponds to increase in the volume of the crystal lattice [15]. Crystallite sizes have been calculated using Scherrer's formula.

3.2 Scanning Electron Microscopy (SEM)

Scanning electron microscopy (SEM) is used to identify the morphology and grain size of the $Mg_{0.5}Zn_{0.5}Fe_2O_4$ prepared by co-precipitation method. SEM graph of $Mg_{0.5}Zn_{0.5}Fe_2O_4$ after sintered at 900 °C are shown in the Fig. 3. From the graphs it is clearly shows that the grains are spherical in shape. The calculated particle size is found to be 40 nm, which is in agreement with the crystallite size calculated from XRD graphs.

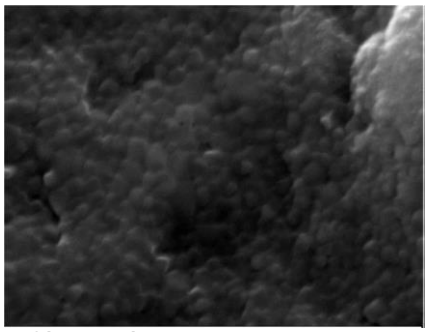

Fig. 3 SEM image of the sintered $Mg_{0.5}Zn_{0.5}Fe_2O_4$

3.3 Thermo Gravimetric Analysis (TGA)/Differential Thermal Analysis (DTA)

From the Fig. 4, TGA graph observed that there is a high weight loss between the temperature range 30-100 °C. It may be because of the evaporation of moisture in the material. Low weight loss is observed between the temperatures 100-400 °C. It may be because of chemical reaction occurred in the $Mg_{0.5}Zn_{0.5}Fe_2O_4$. No significant weight loss is observed from the temperature range 500-1000 °C. It confirms that the formation of phase is completed at 500 °C. It is also confirms the thermal stability of the materials from 500 °C to below its melting point.

From the Fig. 4, DTA graph a sharp endothermic peak is observed between the temperatures ranges 30 °C - 100 °C. It may be because of the evaporation of moisture from the material. Exothermic peak is observed around 400 °C temperature the peak may indicate the crystallization process in the material. No significant peaks observed above 500 °C temperature. It may confirm that the final phase is formed at 500 °C temperature.

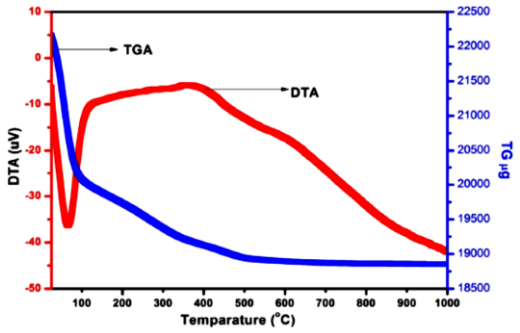

Fig. 4 Schematic diagram of the TGA/TDA

3.4 Dielectric Studies

Fig. 5 displays the frequency dependence of dielectric constant (ϵ) and loss (tan δ) for $Mg_{0.5}Zn_{0.5}Fe_2O_4$ nano composites at room temperature. It can be seen that the dielectric constant decrease steeply at lower frequencies and remain constant at higher frequencies, indicating a usual dielectric dispersion.

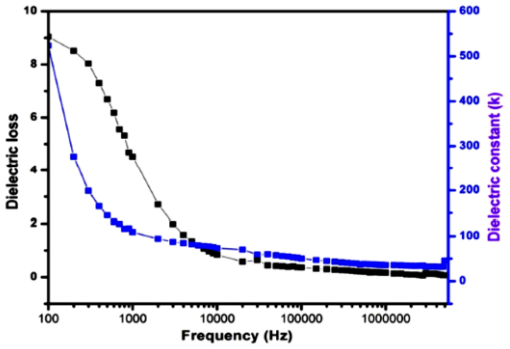

Fig. 5 Shows the dielectric losses of $Mg_{0.5}Zn_{0.5}Fe_2O_4$

The initial decrease in the dielectric constant with frequency up to 80 kHz can be explained by the phenomenon of dipole relaxation. The high value of the dielectric constant at low frequencies may be due to Maxwell–Wagner type interfacial polarization, in agreement with Koop's phenomenological theory. we observed a large difference in ε' between sample low frequency region (1 Hz–80 kHz) and it is attributed to space charge polarization due to the result of the inhomogeneous nature of the dielectric structure in sample . Hence, space charges (carriers located at

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the grain boundary in polycrystalline materials) act as dipoles under an alternating electric field and contribute to polarization. The grain boundary contributions are found to be more effective at low frequencies. Hence, ε' is high for sample whereas it is low for the annealed. In the high frequency region, ε' is almost the same for all samples as the contribution to the dielectric constant arises from electronic and ionic polarizations at high frequencies, which is frequency independent. The variation of the dielectric loss, tan δ, with frequency. In general, the dielectric loss in nanocrystalline ferrites is a result of the lag in polarization with respect to the applied alternating electric field. We observe an abnormal dielectric loss behavior exhibited by sample, which can be due to the resonance effect. When the frequency of the external AC field is equal to the hopping frequency of the charge carriers, the maximum electrical energy is transferred to the oscillating ions and we observe a peak in power loss [16-20].

4. Conclusion

$Mg_{0.5}Zn_{0.5}Fe_2O_4$ nano particles were successfully synthesized by using co-precipitation method. From XRD it has been concluded that single phase spinel structure has been formed at sintering temperature of 900 °C. The particle sizes are in the range of 30 nm to 40 nm calculated from XRD and SEM graphs. The $Mg_{0.5}Zn_{0.5}Fe_2O_4$ powders prepared at low temperature exhibit good crystal structure, fine grain size and dielectric properties. The dielectric constant and dielectric loss of sample have shown good frequency stability and low dielectric losses within the measurement range. Such materials are candidates for capacitor inductor integrating devices such as electromagnetic interference filters in RF communications.

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