

X-RD, TEM, Magnetic studies on $\text{Ni}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ (where $x= 0.2, 0.4, 0.5, 0.6$ and 0.8) Nano Scale Particles by Chemical Co-Precipitation method

B. Suryanarayana^{1a*} V Raghavendra^{1b} K. Chandra mouli^{1c}

¹Solid State Physics and Materials Research Laboratory, Dept.of Engineering Physics,
Andhra University, Visakhapatnam, INDIA

^aE- mail: suryanarayana.badireddi@gmail.com

^bE- mail: raghavendra.vemuri@gmail.com

^cE- mail: ckemburu@yahoo.co.in

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ABSTRACT. Nickel zinc nano particles $\text{Ni}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ (where $x= 0.2, 0.4, 0.5, 0.6$ and 0.8) by Chemical Co-Precipitation method. The samples were characterised by X-ray diffraction (XRD), TEM, VSM. The powders of XRD patterns confirm a single spinel crystalline phase with cubic structure formation with no indication of any other secondary or unidentified phase. The lattice parameter changed from 8.336 Å to 8.382 Å. The average particle size ranged 20 to 80 nm was observed by TEM.

1. INTRODUCTION

Ferrites are well known commercially import functional materials because of their excellent magnetic and electrical properties [1] Among various ferrite materials, the Ni Zn ferrites are considered as the most versatile soft magnetic materials due to their high resistivity and permeability and low eddy current loss for high frequency applications [2, 3]. It is well known fact that properties of ferrites are sensitive to method of preparation, substitution of cations, microstructure, sintering temperature, with general formula AB_2O_4 [4]. Surface mounting devices (SMD) have been extensively developed for electronic applications i.e MLCIs [5]. The main advantage of co precipitation method is expensive, time saving. Absence of ball milling leads to no possibility of loss or gain of material during milling as in case conventional ceramic method. Ni-Zn ferrites are generally used at high frequency due to its low losses; the losses in these materials are also influenced by the grain size. Zinc plays a prominent role in determining the ferrite properties [6]. Gorter [7] carried out the studies on $\text{Ni}_{1-y}\text{Zn}_y\text{Fe}_2\text{O}_4$ ferrite and described the cation distribution in this system. Solid solution precursor technique has been used to prepare fine particle ferrites MF_2O_4 , where $M=\text{Mg, Mn, Co, Ni, and Zn}$ [8] and Ni-Zn ferrites [9] which sinters at low temperature to give 99% theoretical density ferrites. It is reported [10] that zinc ferrite prepared by twin roller quenching method has got, improved fine microstructure and magnetization. Low porosity ferrites are NiZn and LiZn-ferrites [11]. The particle size, significantly affect the magnetic properties and changes in saturation and the transition temperature are found to be due to changes in particle size [12]. Ni-Zn ferrites were prepared to increase sample density by adding SiO_2 and GeO_2 [13]. The Ni Zn Fe_2O_4 silica composite were prepared by sol-gel method different vol % of Ni-Zn ferrite and reported that high resistivity is obtained even at high temperature [14].

2. EXPERIMENTAL PROCEDURE:

In our studies, the (i) NiFe_2O_4 , (ii) ZnFe_2O_4 , (iii) $\text{Ni-Zn-Fe}_2\text{O}_4$ and ferrite compositions were prepared through Co-precipitation technique by using analytical reagent grade, Nickel Chloride (Merck, India), Zinc Chloride (Merck, India), Iron III Chloride (Merck, India), Copper Chloride (Merck, India) and Sodium Hydroxide (NaOH, Merck, India) as starting materials. Fine nanoparticles of ferrites were prepared by co-precipitation method [15]. These chlorides were weighed in the required stoichiometric proportions and dissolved in distilled water and NaOH solution was added to this aqueous solution under continuous stirring at constant speed. The

reaction temperature was maintained constant of 45°C while mixing the alkaline aqueous solution. Therefore, precipitation occurred immediately and the color of suspension changed from brown to dark brown. The mixture was stirred for 2 h at constant temperature of 45°C. The precipitation was washed with distilled water to remove sodium and chlorine ions. The washed samples were dried in air for 20 h. These dried powders were analyzed for phase analysis by powder X-ray diffraction, structural analysis by Transmission electron microscopy and vibration sample magnetometer. X-ray powder diffraction was achieved using Debye-Scherrer formula applying to the FWHM of the (311) peak X-ray diffraction [16]. The dried powders were mixed with 5wt% polyvinyl alcohol and ground well, which were compacted using uniaxial hydraulic pressing machine (with a pressure of 8-9 tonnes) and green bodies (1.7mm in thickness and 6mm in diameter) were sintered at 1000°C for 1 hr with steady rate of heating of 50°C per hour in natural furnace chamber atmosphere to obtain sintered disks. The sintered bodies were cooled naturally along with the furnace. The polished disks were electroded with air dry silver paste by screen printing method. The room temperature dielectric constant, dissipation factor (charge loss or $\tan\delta$) and DC resistivity were characterized as described in the following section.

2.1 Characterisation:

The formation of spinel structure of $\text{Ni}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ (where $x=0.2, 0.4, 0.5, 0.6$ and 0.8) nano ferrite powder was investigated using an X-ray diffractometer PW-1710 using CuK_α radiation with Ni filter at room temperature. Transmission Electron Microscopy (TEM) is a versatile tool capable of characterizing the internal structure of a wide variety of materials [17]. The results obtained from a typical TEM characterization of materials allow a better understanding of the relation between microstructure and properties. In this study, the morphology of fine nanoparticles was observed by Transmission Electron Microscopy (TEM) with a JEOL TEM 1200 microscope. In this study, the sintered and electroded nanoferrite samples were characterized for room temperature dielectric constant (ϵ_{RT}), dissipation factor (charge loss: $\tan\delta$) by Solartron Impedance analyzer (model 1260A). A well-known method for determining the magnetic properties of a wide variety of magnetic systems is vibrating sample magnetometer (EV-7VSM) with this technique the magnetic moment of a sample can be measured with a high accuracy.

Crystallite Size Calculation

The Scherrer formula [18] relates the thickness of the crystallite to the width of its diffraction peaks, and is widely used to determine particle size in ceramics, clays and polymers. The Scherrer formula is given by:

$$D = (k\lambda)/(\beta\cos\theta_B) \quad (1)$$

where, D is the crystallite thickness, β is the broadening of diffraction line measured at half its maximum intensity, k is the shape factor and λ is the wavelength of the X-ray beam.

3. RESULTS AND DISCUSSION

3.1. X-ray diffraction (XRD)

Fig.1. shows XRD patterns confirmed the single spinel crystalline ferrite phase with cubic structure ($m3m$) formation and with no indication of any other secondary or unidentified phase or incomplete reaction or impurity peaks. It is found from XRD figure that the intensities of XRD peaks of the spinel Cubic ferrite phase gradually increase with the increase of x throughout the series. The observed reflection pattern matches with JCPDS File no.08-0234($\text{NiZnFe}_2\text{O}_4$). The calculated lattice parameter 'a' of ZnFe_2O_4 is 8.329 Å, NiFe_2O_4 is 8.331 Å and as x varied from 0.2 to 0.8, the lattice parameter changed from 8.336 Å to 8.382 Å, respectively in the series of $\text{NiZnFe}_2\text{O}_4$ ferrites [19]. The average value of crystallite size calculated for ZnFe_2O_4 , NiFe_2O_4 and x varied from 0.2 to 0.8 are ranged from 17 to 38 nm, respectively in $\text{NiZnFe}_2\text{O}_4$ ferrites series [20].

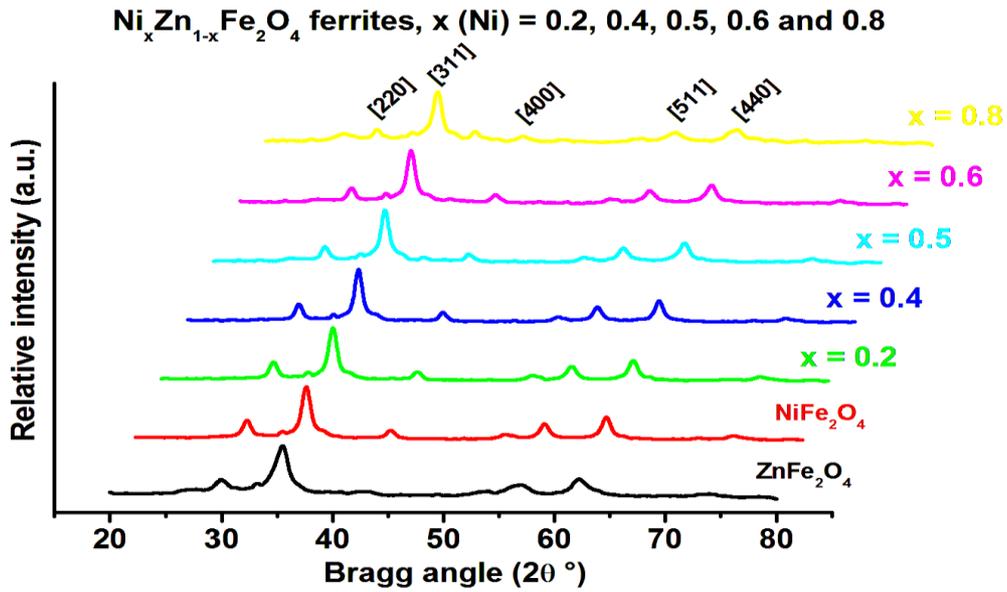


Fig 1: Powder X-ray diffraction patterns of $\text{Ni}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$

3.1 Microstructural analysis:

In our study, the TEM images indicate that most of the particles appear almost spherical in shape; however, some elongated particles are also present in the images. Some moderately agglomerated particles as well as separated particles are also present in the ferrite powders. In this study, the TEM pictures reveal that co-precipitation technique is one of the best chemical routes to obtain fine agglomerate free ferrite nano particles. According to literature, the average particle size of 25 ± 5 nm was observed in the NiFe_2O_4 ferrite system by TEM [21, 22]. It is observed that average particle size ranged 20 to 80 nm in the above series.

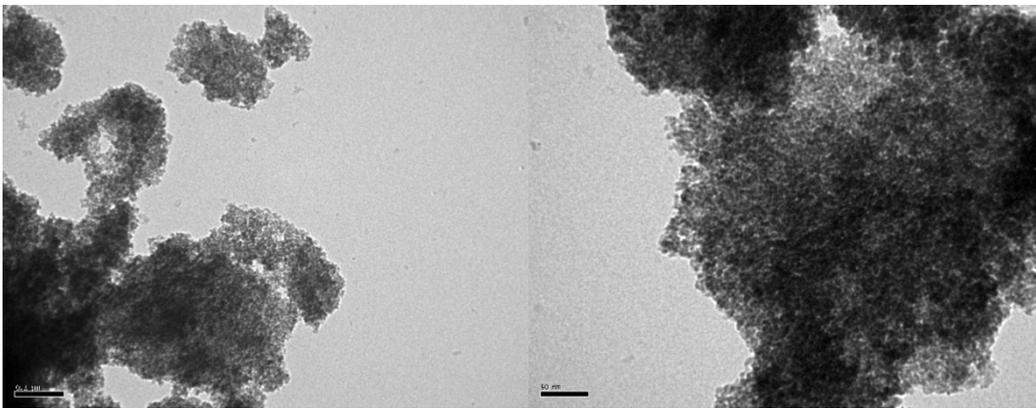


Fig 2: Tem picture of ZnFe_2O_4

Fig 3: Tem Picture of NiFe_2O_4

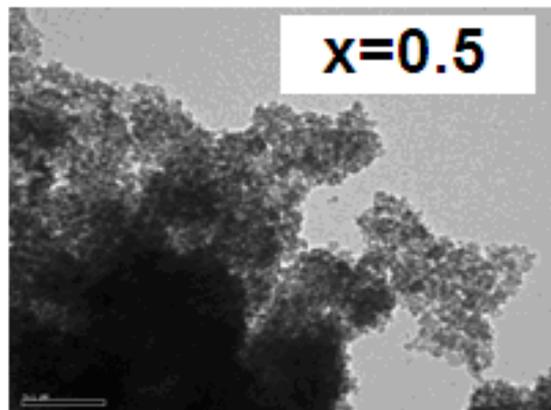


Fig 4: Tem picture of $\text{Ni}_{0.5}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$

3.2 .Magnetic Properties:

The magnetic hysteresis loops of nanoferrites were investigated at room temperature signifies the existence of an ordered magnetic structure in the spinel Cubic ferrite system. The increase in magnetization could be due to the fact that the individual ferrite grains act as centres on account of polarization which may be caused by the cationic vacancies. It is well known that the saturation magnetization of a spinel ferrite largely depends on its composition and particle size, while the coercive field depends on composition, particle size and shape [23, 24, 25]. In this study, a saturation magnetization (M_s) in the range of 7.94 to 8.76 emu/g with a range of (+5000 Oe) to (-5000 Oe) of coercivity were observed. Hysteresis curve of $Ni_xZn_{1-x}Fe_2O_4$ are shown in Fig 5. Nanoferrites and thus influenced the magnetic properties. Thus, our results are in accordance with the studies presented in the literature [26, 27].

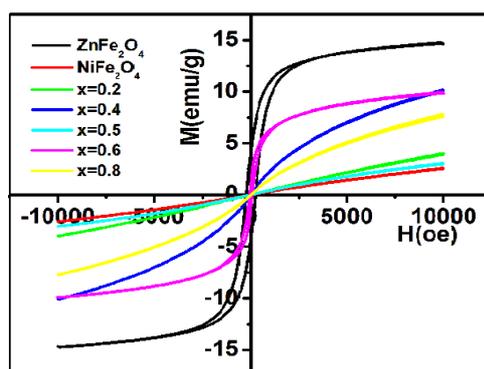


Fig 5: Hysteresis curve of $Ni_xZn_{1-x}Fe_2O_4$

4. CONCLUSIONS

$Ni_xZn_{1-x}Fe_2O_4$ (where $x = 0.2, 0.4, 0.5, 0.6$ and 0.8) Nano Scale Particles prepared successfully through by Chemical Co-Precipitation method. All the samples showed single phase cubic spinel structure and the lattice parameter changed from 8.336 Å to 8.382 Å. The average value of crystallite size ranged from 17 to 38 nm. TEM images indicate that most of the particles appear almost spherical in shape and the average particle size ranged 20 to 80nm observed. In Hysteresis study saturation magnetization (M_s) in the range of 7.94 to 8.76 emu/g with a range of (+5000 Oe) to (-5000 Oe) of coercivity were observed.

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