ISSN 2229-5518

Synthesis of Ni-Zn Ferrites Using Low Temperature Sol-Gel Process

Pushpendra Kumar, Pawan Mishra, Sanjay Kumar Sahu

Abstract— Quality of ferrite materials have dominant role in the performance of ferrite devices. Properties of ferrites are found to be dependent on their chemical composition and microstructure, which in turn are governed by the preparation process. In our work we prepared five different compositions of nickel-zinc ferrites by using low temperature sol-gel technique Structural analysis of prepared samples were done by using X-ray differaction, Field emission scanning electron microscopy and Transmission electron microscopy.

Index Terms— Composites, Ni-Zn ferrites, Nanomaterials, Sol-gel process, Structural Analysis,

1 Introduction

Nanomaterials with an average grain size in the range of 10 to 20 nm have attracted research interest for more than a decade since their physical properties are greatly influenced by cotrolling the material at atomic scale. Nickel-zinc ferrites are magnetic materials of immense technological importance with diverse applications such as low and high frequency transformer cores, antenna rods and microwave devices [1]. Quality of ferrite materials have dominant role in the performance of ferrite materials besed devices. Properties of ferrites are found to be highly sensitive to their chemical composition and microstructure, which in turn are governed by the preparation process.

In our work we achieved enhancement in the structural, magnetic and transport properties of the Ni-Zn ferrite. In this paper we are reporting the simple route for synthesis of Ni-Zn ferrites and structural analysis of the prepared samples.We studied change in properties with respect to composition of Zn^{+2} ion in nickel ferrite, frequency and temperature. High resistivity of prepared sample using the sol-gel process established in our study which is a pre-requisite for ferrites operating at high frequencies to curb the eddy current losses. Detailed analysis of magnetic and electrical properties will be published in next manuscript.

2 Sample Preparation

The properties of ferrites are affected by the microstructural problems which have become the most serious obstacles in obtaining high quality reproducible ferrites [2]. So in this way there are so many processes to synthesize the nano magnetic materials but some of them have some inherent drawbacks such as poor compositional control, chemical inhomogeneity and introduction of various impurities. From the literature survey on various synthesis methods it can be said that chemical methods have overcome these problems [3].

The chemical methods such as co-precipitation [4], citrate precursor method [5], hydrothermal synthesis [6] as well as the sol–gel [7] process have been selected to overcome the problem in improving the performance of the ferrites. Among these methods, the sol–gel preparation is more preferable due to its numerous advantages. Some advantages which were evident in this study were its high purity, chemical homogeneity, small and uniformed particle sizes, energy saving, no reaction with containers which increase purity.

Ni-Zn ferrites of composition $Ni_{1-x}Zn_xFe_2O_4$ with x = 0.0, 0.25, 0.35, 0.50, 0.65, 0.75, 1.0 have been prepared. For this Zinc acetate dihydrate $Zn(COO)_2 .2H_2O$ and Nickel nitrate hexahydrate $Ni(NO_3)_2.6H_2O$ were mixed with the expected molar ratio (doping concentration). In our experiments, we took 0.05 mol zinc acetate dihydrate. The mixture of the two salts was then dissolved by 4 ml ethylenglycol (liquid) and five to seven drops of glycerol (to stablize and increase the solubility of the solution) under magnetically stirring at 175°C for 10 minutes. Then we obtain a transparent homogenous sol. Then we cool down the above solution and dilluted it by adding 10 ml isopropanol (2-propanol). We used triethylamine (TEA) as the catalyst for the sol-gel process. We then increase the stirring rate and gradually 5ml triethylamine is dropped in the solution at room temperature. Once finishing the triethylamine dropping, we again stir the solution for 15 minutes further. From this solution we can obtain nanoparticles by heating the sol at 150°C in air for 3 hrs and until completely smaller gel particles are formed. These dry gel particles were calcined at 350°C for 4h to obtain the phase. The powder samples were studied using XRD to determine the structural phase of the prepared materials.

- *Authors of this paper are Alumni of Indian Institute of Technology, Kharagpur and currently working as Faculty Members in Department of Electronics and Communication, The ICFAI University, Dehradun, India,*
- *Corresponding Author:*
  Pawan Mishra, E-mail: pawanmishra2010@gmail.com

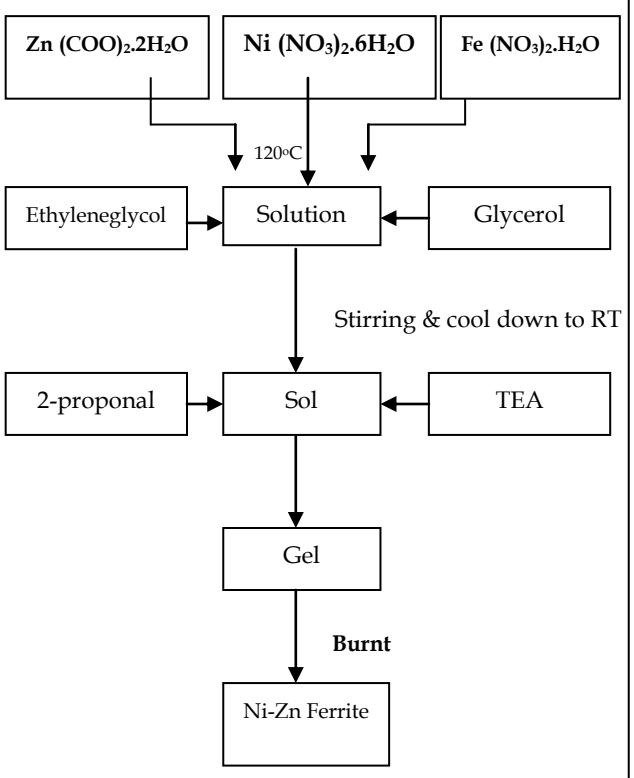


Fig.1. Flow chart for synthesis the powder Ferrite via sol-gel method

3 Results and Discussion

X-ray powder diffraction technique has been used to carry out phase analysis of prepared ferrite samples ($Ni_{1-x}Zn_xFe_2O_4$ where x= 0.0, 0.25, 0.35, 0.5, 0.625, 0.75, 1) in polycrystalline form by sol-gel method. Initially the sample were sintered for 4 hours at 350^{0}C for making a homogenous product and XRD patterns were taken by Rigaku XRD miniflex, diffractometer equipped with Co Kα (λ= 1.67A^0) radiation source. The samples were through an angle of 20-70^0 at a constant scanning speed to identify the phases formed. The diffraction patterns were taken out and plotted with the help of origin software, which are analyzed to calculate d (inter-atomic spacing) and to index (h, k, l) using JCPDF (joint committee on powder diffraction standards) data [8]. The XRD data for all the ferrite samples are shown in fig.3.1. We notice that the positions of the peaks are similar to the reported values earlier [9]. The XRD patterns of both compositions agree well with the standard JCPDS data, clearly indicate their single phase and formation of spinel structure. The XRD data for all the ferrite samples are shown in fig.3.1. We notice that the positions of the peaks are similar to the reported values earlier [9]. The XRD patterns of both compositions agree well with the standard JCPDS data, clearly indicate their single phase and formation of spinel structure.

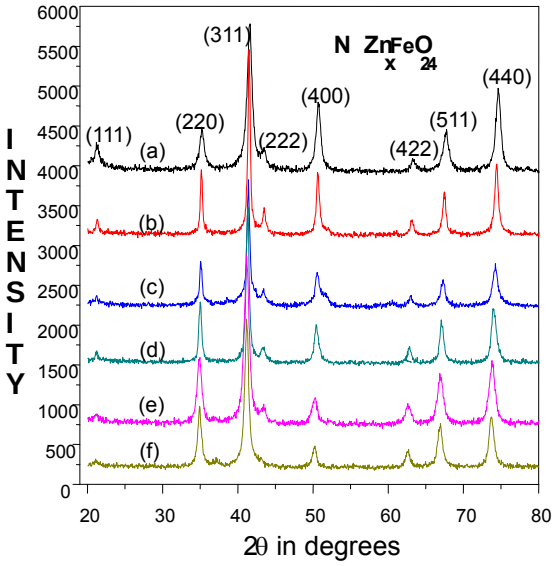


Fig.2. The X-ray diffraction patterns for the samples $Ni_{1-x}Zn_xFe_2O_4$ for **x**, (a) 0.0, (b) 0.25, (c) 0.35, (d) 0.50, (e) 0.75, (f) 1

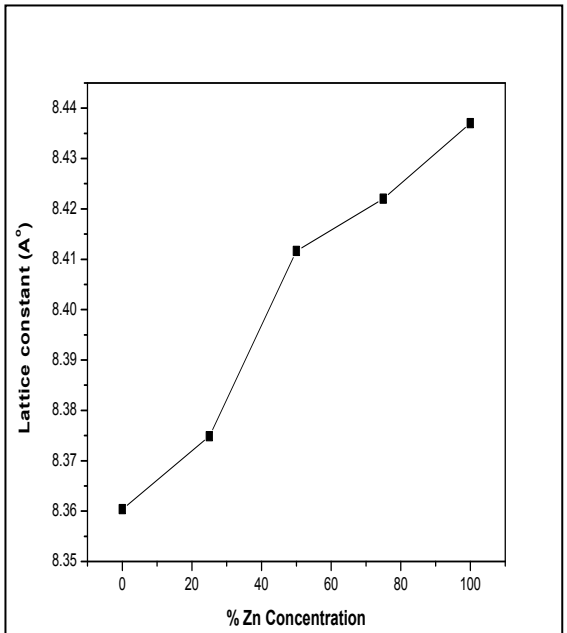
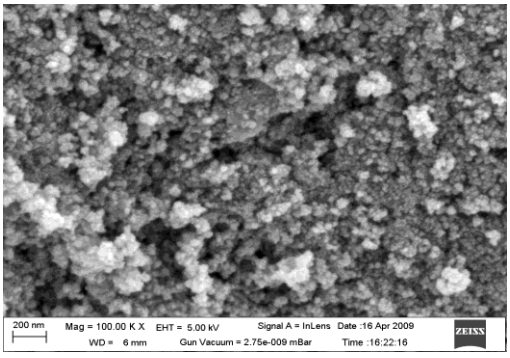
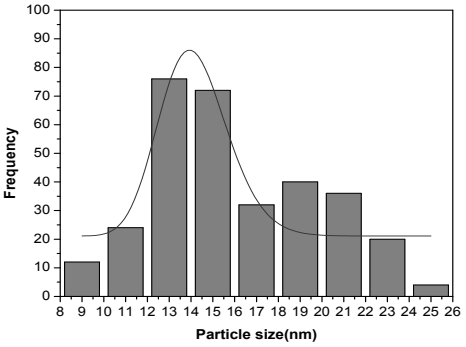
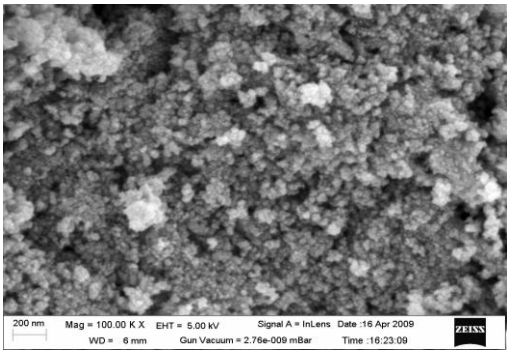


Fig.3. Compositional variation of the lattice constant for $Ni_{1-x}Zn_xFe_2O_4$







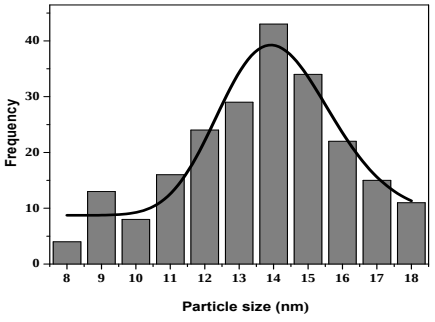
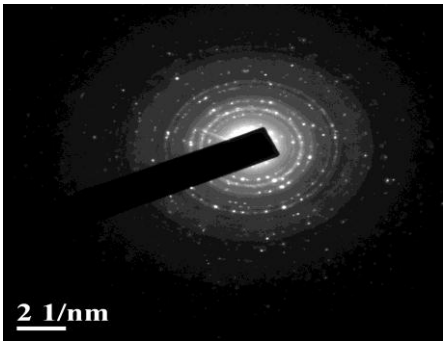
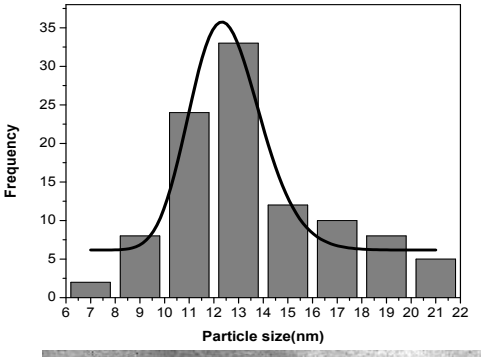
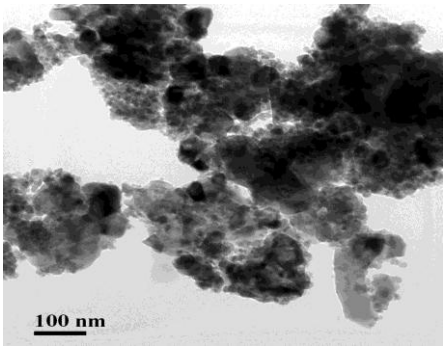


Fig.4. Field emission micrograph and histogram of (a) $Ni_{0.5}Zn_{0.5}Fe_2O_4$ (b) $Ni_{07.5}Zn_{0.25}Fe_2O_4$.







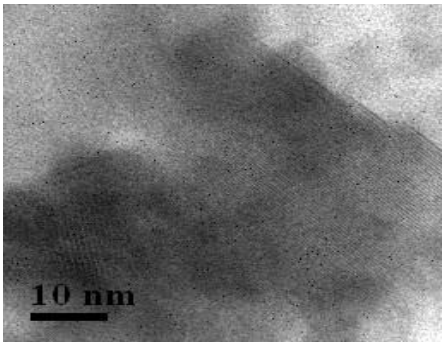


Fig.5. TEM micrograph of $Ni_{0.75}Zn_{0.25}Fe_2O_4$

The lattice parameter for different composition of this series have been calculated using the value of d-spacing, which were calculated by using Bragg law for each peak and each sample, was calculated using the formula

$$a = d_{hkl}\sqrt{h^2 + k^2 + l^2}$$

Where h, k, l are the miller indices of the crystal planes. The values of lattice parameter as a function zinc concentration are plotted in fig.2 for various $Ni_{1-x}Zn_xFe_2O_4$ ferrites.From the fig.2, we can see as we are increasing the Zn^{+2} ion i.e. x value increasing peaks are shifting towards left from (a) to (e) i.e. θ is decreasing or may be say d increasing and from the above formula a will also increase. The value of the calculated lattice constant for different Zn concentrations are given in fig.3, the lattice constant is seen to increase linearly with Zn content for the composition $Ni_{1-x}Zn_xFe_2O_4$. From the XRD data it is clear that, Ni-Zn ferrite system has a cubic spinel configuration with unit cell consisting of eight formula units of the form $[Zn_xFe_{1-x}]^A[Ni_{1-x}Fe_{1+x}]^BO_4$, where A and B represent tetrahedral and octahedral sites respectively. The Ni^{2+} ions have a marked preference for octahedral sites because of their favorable fit of charge distribution of this ion in the crystal field of the octahedral site whereas Zn^{2+} ions have preference for the tetrahedral site due to their readiness to form covalent bonds involving sp^3 hybrid orbitals. The observed linear increasing of lattice constant with Zn content can be attributed to the large ionic radius of Zn^{2+} (0.84A˚) as compared to the ionic radius of Ni^{2+} (0.74 A) [10-11]. The variation of the lattice parameter as a function of Zn ions in the $Ni_{1-x}Zn_xFe_2O_4$ matrix follows the Vegard's law [12].

The microstructure and compositional analysis of all the Ni-Zn ferrite sample's pallets were investigated with a high resolution Carl Zeiss SMT Ltd SUPRA 40 Field emission scanning electron microscope (FESEM). Here, before taking the pattern for morphology, gold coating is necessary because ferrite is high resistive material. By gold coating we can avoid the charging effect.
The morphology of grain structure as seen by field emission scanning electron microscopy is uniform and nearly spherical as shown in fig.4. With the help of software we plotted the histograms between number of particles and diameter of the particles. Histogram shows that the particle size is in the nanometric (11-15nm) region, good agreement with values obtained from the XRD data. As can be seen the, microstructure consists of relatively small size grains. It is noted that small grains are preferred in ferrites, as oxidation advances faster in smaller grains thus leading to the acceleration of the Fe^{2+} to Fe^{3+} transformation.

The particle size and morphology of all NI-Zn ferrite samples were investigated by transmission electronic microscopy (TEM, model: H-800, HITACHI, Japan). For the TEM observations, powders were supported on carbon-coated copper grids which was ultrasonically dispersed in ethanol

The TEM photographs of $Ni_{0.75}Zn_{0.25}Fe_2O_4$ particles obtained by the sol-gel method are shown in fig.5. The ferrite particles had nanosize spherical morphology and uniform size. The particle sizes were determined from the TEM to be 12–14 nm. These values are in agreement with those calculated from XRD peaks.

4 Conclusion

The use of ferrites has become established in many of telecommunication and electronic engineering and they now embrace a very wide diversity of compositions, properties and applications. Therefore, the effects of the process variables on the magnetic and electrical properties of the finished ferrite pieces have been a subject of great importance. Most important factors in any manufacturing process are the economy of the process and quality of the product. This generally means that properties are enhancing but the manufacturing cost reducing, than it will be acceptable. Ni-Zn ferrite samples of nanometer size with different composition have been prepared using low temperature sol-gel method. X-ray diffraction indicates the formation of Ni-Zn ferrite, as single phase, on 350^0C sintered and for x variation found the shifting among the peaks due to variation of the d value by Zn^{+2} ion concentration. The structural properties of ferrites show that the lattice constant increasing linearly with Zn^{+2} ion concentration due to difference in ionic radius. Results from the FESEM and TEM are in well agreement with XRD data.

5 Refrences

[1] I.H. Gul, A.Z. Abbasi, F. Amin, M. Anis-ur-Rehman, A. Maqsood, J. Magn. Magn. Materials 311 (2007) 494.
[2] A. Thakur, M. Singh, Ceram. Int. 29 (2003) 505.
[3] A. Verma, T.C.Goel, G.mediratta, R.G.Gupta, J. Magnetic Mater. 192 (1999) 271.
[4] E. Auzans, D. Zins, R. Massart, J. Mater. Sci. 34 (1999) 1253.
[5] T. Upadhyay, R.V. Upadhyay, R.V. Mehta, V.K. Aswal, P.S. Goyal, Phys. Rev. B 55 (1997) 5585.
[6] M. Rozman, M. Drofenik, J. Am. Ceram. Soc. 78 (1995) 2449.
[7] D. Makovec, A. Kosak, M. Drofenik, Nanotechnology 15 (2004) 160.
[8] P. Bayliss, D.C. Erd, M.E, More Annp Sabina and D.K. Smith Mineral Powder Diffraction file, JCPDF, USA (1986).
[9] A.K.M.A. Hossain et al. journal of magnetism and magnetic materials 312 (2007) 210-219
[10] A.M. EI-sayed ceramic international 28 (2002) 363-367.
[11] S.M. Hoque, Md.A. Choudhury, Md.F. Islam, J. Magn. Magn. Mater. 251 (2002) 292.
[12] Komar AP, Bull Acad Sci USSR Ser Phys (1954) 18:122.