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Synthesis and Characterization of Nickel Doped Zinc Ferrite Nanoparticles

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Abstract. Nickel doped zinc ferrite compounds with the formula $Ni_xZn_{1-x}Fe_2O_4$($0.0 \leq x \leq 0.6$) were prepared using sol gel technique. The influence of the Ni content on the lattice parameter 'a', stretching vibrations, line width, line intensity and the magnetization of zinc ferrite samples were subsequently studied. The X-ray diffraction patterns confirm the synthesis of single crystalline $Ni_xZn_{1-x}Fe_2O_4$ ferrite nanopaticles. The lattice parameter decreases whereas crystallite size increases with the increase in nickelionconcentration. Electron paramagnetic resonance spectra was used to evaluate lande's splitting factor (g value), peak to peak line width (ΔH_{p-p}), spin concentration (N_s) and relaxation time (τ_s). Line width, spin concentration increases with Ni^{2+} ions doping for (x = 0, 0.2, 0.4) and then decreases for x = 0.6, whereas the g value shows the reverse trend for the same composition. Magnetic parameters such as saturation magnetization (M_s), remanence (M_r) and coercivity (H_c) were measured by search coil method. The value of saturation magnetization increase from 19.94 emu/gm for x = 0.0 to 58.77emu/gm for x = 0.6.
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1. INTRODUCTION

The nanomagnetic materials have remarkable interest owing to their interesting properties and a wide variety of applications. The magnetic ferrite nanoparticles are very important because of their applications in the fields of high-density magnetic media, recording color imaging, ferrofluids, high-frequency devices and magnetic refrigeration [1, 2].These nanoparticles have special properties as compared to the bulk because of the large surface to volume ratio which in turn is responsible for their several unusual properties like spin canting, surface anisotropy, superparamagnetism etc. The doping of nickel ions in spinel structure of zinc ferrite results in modification of basic electrical, magnetic and microstructural properties. In the present work different concentrations of Ni^{2+} ions are substituted in the lattice of zinc ferrite to understand the effect on the structural and magnetic properties of zinc ferrite. These samples were prepared by sol-gel method.

2. EXPERIMENTAL

The starting materials used were AR grade $Ni(NO_3)_2.6H_2O$, $Zn(NO_3)_2.6H_2O$and $Fe(NO_3)_3.6H_2O$.Equimolar solutions of these salts were prepared in ethylene glycol in stoichiometric ratio and homogenized at 60 °C by using magnetic stirrer. After that the solution was heated at 80 °C until gel formation takes place. The gel was then heated at 100 °C overnight in the furnace to obtain the desired material in powder form. This powder was grounded by using mortar and pestle andwas used for further characterizations.The X-ray diffraction patterns (XRD) were recorded using a Rigaku powder X-ray diffractometer with Cu $K_\alpha$radiations. The shape, size and distribution of these ferrite nano-particles were studied with the help of high resolution transmission electron microscope (model TECNAI F30).The magnetic measurements were carried out by search coil method. The EPR measurements were carried out at ambient temperature by using X- band EPR Spectrometer Model: A300 Make: BrukerBiospin, Germany.

3. RESULT AND DISCUSSION

The XRD patterns of as synthesized samples with various nickel and zinc composition are shown in figure 1.X-ray diffraction patterns shows broad peaks indicating small crystallite size and ultrafine nature of the particles. All the diffraction peaks matches perfectly to the JCPDS card no. 19-0629. The XRD pattern shows that peaks shift towards higher angle side with increasing nickel ion concentration. Also the peak intensity is decreasing with increase in nickel content.

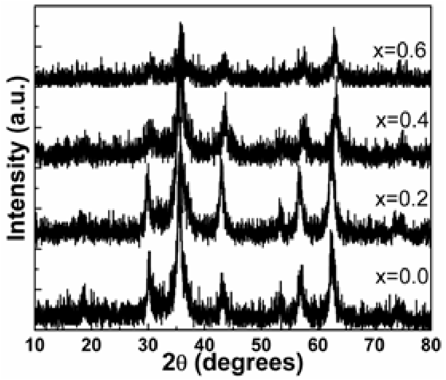

FIGURE 1: XRD pattern of $Ni_xZn_{1-x}Fe_2O_4$
(Where x =0.0, 0.2, 0.4, 0.6) samples

The lattice parameter for the spinel phases was computed and it is found that lattice parameter decreases with the increase in Ni^{2+} ion concentration. The decrease in the value of lattice parameter is attributed to the smaller ionic radii of Ni^{2+} ions as compared to Zn^{2+}cations [3, 4].The average crystallite size (D) was calculated from the peak broadening using Scherrer's formula.

$$D = 0.89\ \lambda / \beta \cos\theta$$

Where D is the Crystallite size, λ is wavelength of X-rays, β is full width at half maximum (FWHM) measured in radians and θ is the Bragg's angle. The values of average crystallite size, lattice parameter calculated from XRD patterns are listed in table 1.

TABLE 1: XRD analysis of $Ni_xZn_{1-x}Fe_2O_4$ (where x= 0, 0.2, 0.4, 0.6) nanoparticles

Samples	Lattice parameter (Å)	Crystallite size (nm)
$Ni_{0.0}Zn_{1.0}Fe_2O_4$	8.368	10
$Ni_{0.2}Zn_{0.8}Fe_2O_4$	8.331	12
$Ni_{0.4}Zn_{0.6}Fe_2O_4$	8.287	16
$Ni_{0.6}Zn_{0.4}Fe_2O_4$	8.264	19

High resolution transmission electron microscopy (HRTEM) has provided further insight into the morphologies and structure detail of these nickel zinc ferrite sample. Fig. 2 shows the HRTEM image of $Ni_{0.2}Zn_{0.8}Fe_2O_4$ sample. The size of these ferrite nanoparticles calculated from HRTEM images are consistent with the size calculated by Debye-Scherrer formula. The average size distribution of the particles was about 09, 13, 17 and 20 nm for x= 0.0, 0.2, 0.4 and 0.6 concentrations respectively.

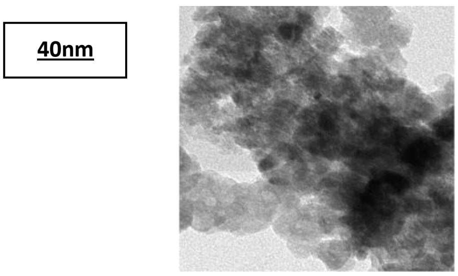

FIGURE 2: HRTEM of $Ni_{0.2}Zn_{0.8}Fe_2O_4$ sample

From the magnetic measurement, it is observed that that the substitution of nickel ions increased the domain magnetization of the samples. The increase in saturation magnetization with nickel content is due to many factors like cation distribution, the existence of surface spin or the formation of spin glass structure etc. The increase in saturation magnetization is also due to the larger magnetic moment of nickel ions as compared to zinc ions. The smaller value of saturation magnetization for samples with less nickel content may be due to lattice defects, weaker magnetic superexchange interactions between A-sites and B-sites. The saturation magnetization increase from 15.35emu/gm to a value of 45.47emu/gm as the Ni^{2+} ion concentration increases from 0 to 0.6.These super paramagnetic Ni-Zn ferrite nanoparticles can be used for ferrofluid applications.

4. CONCLUSIONS

The x-ray diffraction patterns confirms the synthesis of single crystalline phase of $Ni_xZn_{1-x}Fe_2O_4$ (x=0, 0.2, 0.4 and 0.6) nanoparticles.The lattice parameter decreases with the increase in nickel content, resulting in reduction in lattice strain. The magnetization measurement shows the superparamagnetic nature of $Ni_{0.0}Zn_{1.0}Fe_2O_4$ and$Ni_{0.2}Zn_{0.4}Fe_2O_4$ samples whereas the material with x= 0.4, 0.6 shows ferromagnetic nature with increase in remanence and coerecivity.

5. REFERENCES

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