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Preparation, characterization and magnetic properties of nanocrystalline Ni-Zn ferrites

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Abstract

Nanocrystalline $Ni_xZn_{1-x}Fe_2O_4$ *ferrites (with x = 0.1,0.3,0.5,0.7,0.9) were synthesizes by sucrose process.The powders were sintered at 900* ^0C *for 12 h to densify properly.The samples were characterized by XRD, TEM and IR spectroscopy. XRD confirms single phase spinel structure. The lattice constant increases with increase in Zn content. TEM reveals nanocrystalline nature of samples while IR absorption spectra shows two prominent bands for characteristic A and B site vibrations. The magnetic properties were measured by using high field hysterisis loop tracer.*

Key words: Nanocrystalline ferrites, sucrose method, TEM, IR spectra, magnetic properties.

INTRODUCTION

Nanacrystalline spinel ferrites have been investigated in the recent years by many researchers. These ferrites are materials of interest because of their unique electric, dielectric and magnetic properties. Ni-Zn ferrite show good magnetic properties for technical applications hence these are widely used in magnetic cores of read-write heads for high speed digital recording [1]. Magnetic properties of Ni-Zn, Mn-Zn have attracted attention because of their use at high frequency applications [2]. Ni-Zn ferrites are used at high frequencies due to their high resistivity and low eddy current loss [3]. It has low coercivity and saturation magnetization[4]. It also used as core material for transformers.

In recent years number of physical and chemical techniques have been developed to prepare nanosized magnetic materials. The widely used chemical methods are electro deposition [5], sol gel route [6], co-precipitation [7], micro emulsion technique [8], Glyoxylate precursor method [9], hydrothermal method [10], reverse micelle technique [11], sucrose method [12, 13].

The sucrose process is easy, cost effective solution based method for preparation of nanoferrite powders at lower processing temperatures.

MATERIALS AND METHODS

2.1 Preparation

Initially AR grade metal nitrates viz. nickel nitrate (Ni $(NO_3)_2 6H_2O$) zinc nitrate (Zn $(NO_3)_2$ $6H_2O$) and iron nitrate (Fe $(NO_3)_3 9H_2O$) were taken in desired stoichiometric proportions and dissolved in distilled water. 10% aqueous solution of polyvinyl alcohol (PVA) and some sucrose were added to this solution followed by thorough stirring and heating at 80 ^{0}C for about 30 minutes. Resulting solution was heated at about 200 ^{0}C to remove NO_2 fumes completely. This gives black, fluffy, voluminous organic based precursor material. It was then ground to fine powder and presintered at 800 ^{0}C for about 9h.The presintered powders were subjected to hand milling, sieved and then was palletized by using hydraulic press. The pellets were finally sintered at 900 ^{0}C for 12h in air to densify properly.

Here when aqueous solution of sucrose is mixed with nitrates, sucrose gets hydrolyzed to gluconic acid and polyhydroxy acid. The gluconic acid has carboxylic acid group and fixed linear hydroxyl groups. Because of this, it can participate in to metal ions and form a polymer chain with PVA.

Thus sucrose provides wrapping through co-ordination for the cations in solution and circumvents their selective precipitation from the solution during evaporation process. It also provides the efficient fuel to combustion reaction being oxidized by the nitrate ions. The PVA solution provides polymeric matrix structure and also provides heat for combustion of carbonaceous materials.

2.2 Characterization

After sintering phase analysis of samples was done by using X ray diffractometer (Philips model PW 3710 EX), The transmission electron microscope (TEM model JEOL- 1200 EX) was used to study the microstructure. IR spectrographs of samples were obtained by using Perkin Elmer IR spectrometer (model 783) in wave number range 350 cm^{-1} to 700 cm^{-1} in KBR medium. High field hysterisis loop tracer was used to measure saturation magnetization.

RESULT AND DISCUSSION

3.1 XRD, Microstructure and IR absorption

The XRD patterns of all samples after final sintering were taken in 2θ range 20^0 to 80^0 were shown in Fig. 1.

The prominent line in diffraction pattern for all samples corresponds to (311) plane and no extra peaks were observed confirming formation of single phase cubic spinel structure. The Zn^{2+} have larger ionic radius (0.83 ^{0}A) as compare to Ni^{2+} (0.74 ^{0}A) and Fe^{3+} (0.65 ^{0}A) ions.

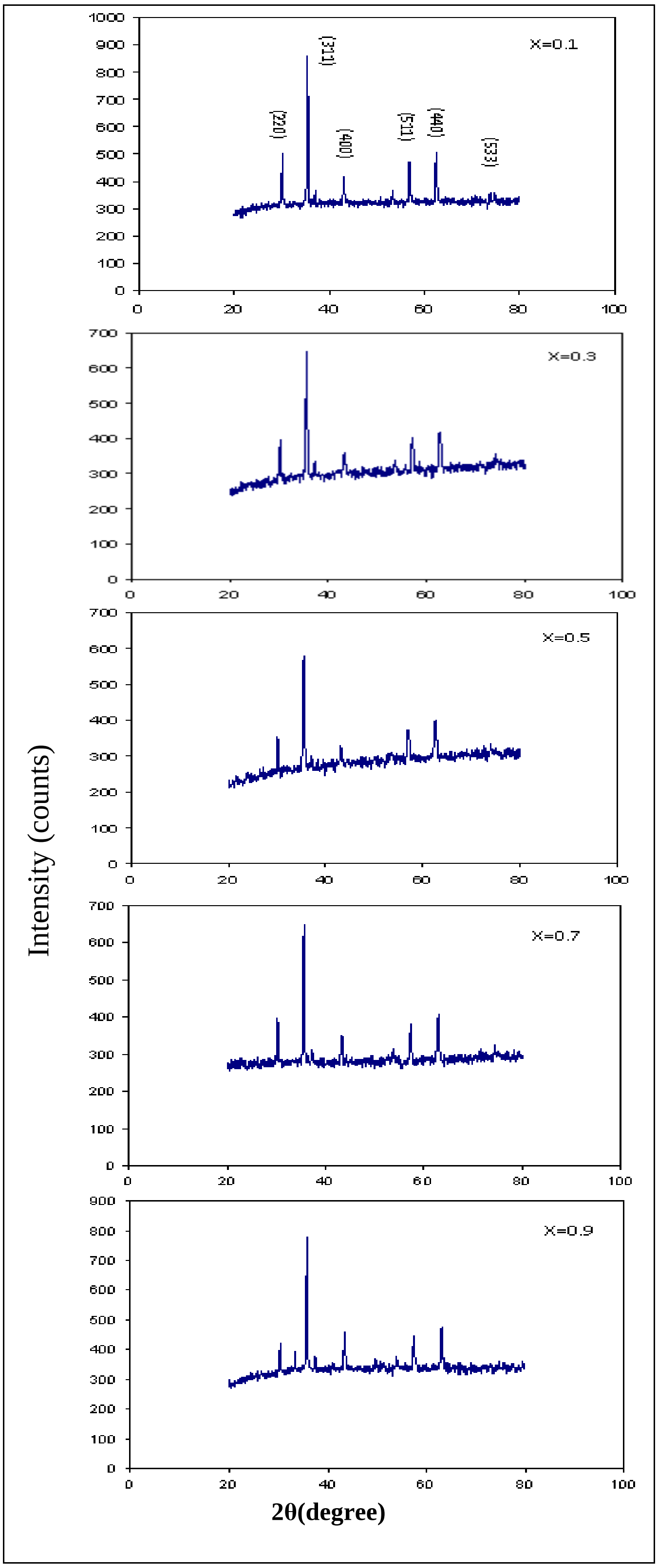

Fig. 1 XRD patterns of $Ni_xZn_{1-x}Fe_2O_4$ ferrites

The lattice parameter ‘a’ is very sensitive to Zn^{2+} ion concentration. So ‘a’ increases with Zn^{2+} content by obeying Vegard’s law [14]. Similarly the other parameters viz. cation radii r_A, r_B and the bond lengths R_A, R_B also increases with Zn^{2+} content due to increase in volume of unit cell. The values are listed in Table-1.

Table 1. Data on lattice parameter, cation radii and bond length, absorption band ν_1, absorption band ν_2 saturation magnetization and Y-K angles for $Ni_xZn_{1-x}fe_2O_4$ nanoferrites

Parameter	x=0.1	x=0.3	x=0.5	x=0.7	x=0.9
a (^{0}A)	8.427	8.390	8.364	8.356	8.342
$r_A(^{0}A)$	0.592	0.582	0.576	0.574	0.571
$r_B(^{0}A)$	0.689	0.680	0.674	0.672	0.668
$R_A(^{0}A)$	1.941	1.932	1.926	1.924	1.921
$R_B(^{0}A)$	2.041	2.032	2.025	2.023	2.020
$\nu_1(cm^{-1})$	416.94	417.94	416.90	416.92	416.72
$\nu_2(cm^{-1})$	559.06	577.93	577.46	583.42	587.28
M_s(emu/gm)	3.07	21.50	43.68	46.00	38.19
Y-K angle	$17^{0}25'$	$58^{0}31'$	$46^{0}11'$	$29^{0}11'$	0^{0}

Fig. 2 TEM micrographs of nanocrystalline $Ni_XZn_{1-X}Fe_2O_4$ ferrites

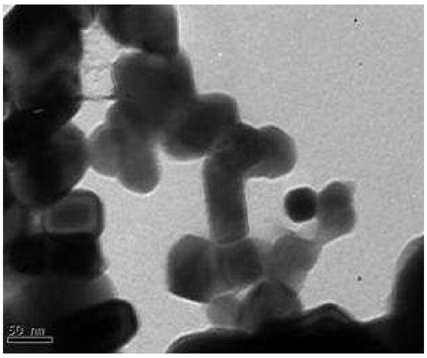

Fig.2 represents Transmission electron micrograph of sample. It shows nearly uniform spherical ferrite particles having particle size between 30 nm to50 nm. This is lower than the particle size of nanoferrites prepared by other chemical method[15]. It is also seen that variation in Zn content does not affect on particle size.

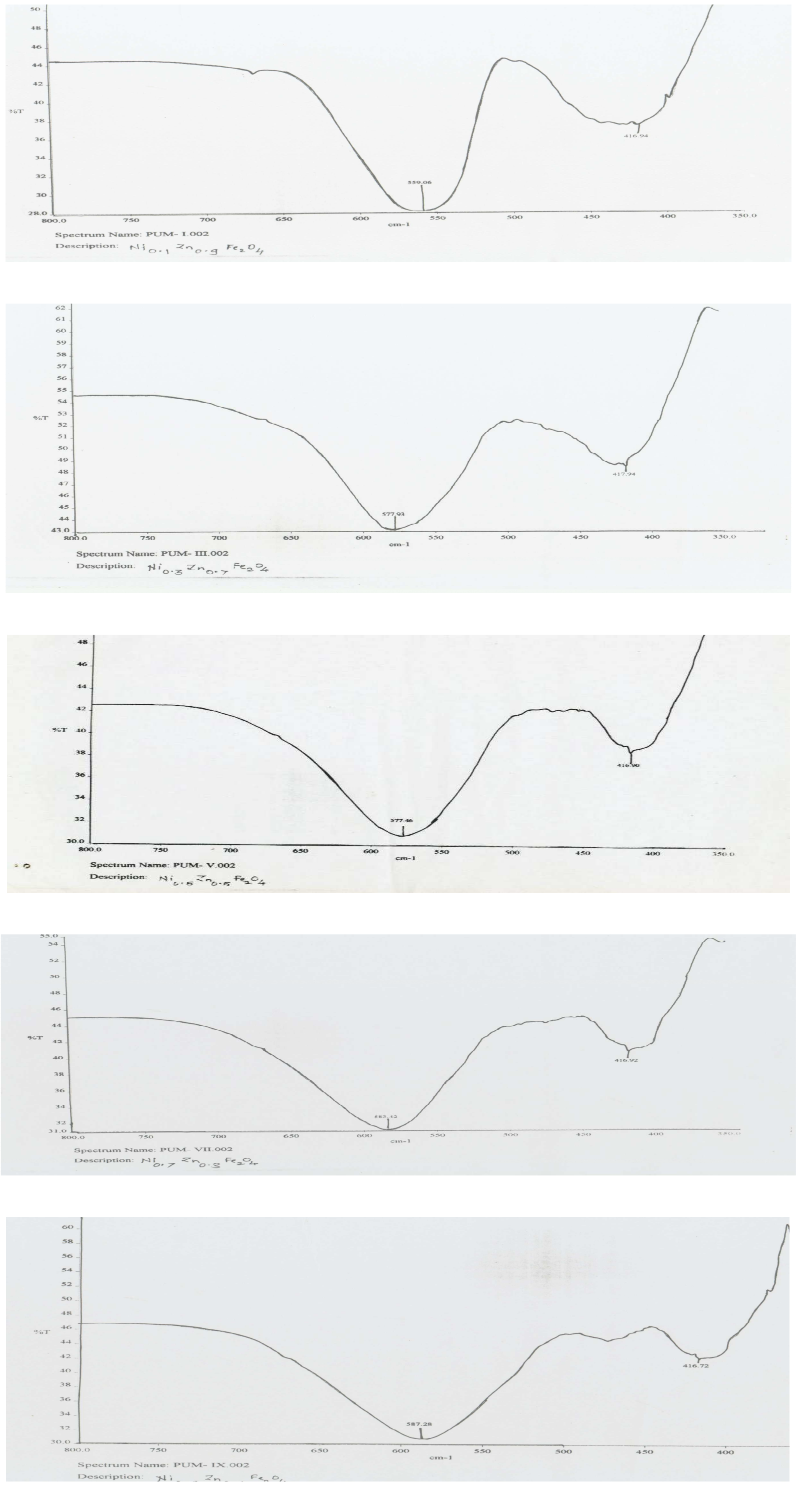

Fig. 3 IR spectrographs of nanocrystalline $Ni_XZn_{1-X}Fe_2O_4$ ferrites

Fig.3 shows IR spectrographs of all samples. It provides information about positions of ions in crystal lattice through crystal vibrations. It shows two prominent absorption bands with vibrations υ_1 and υ_2 around 600 cm^{-1} and 400cm^{-1} respectively.

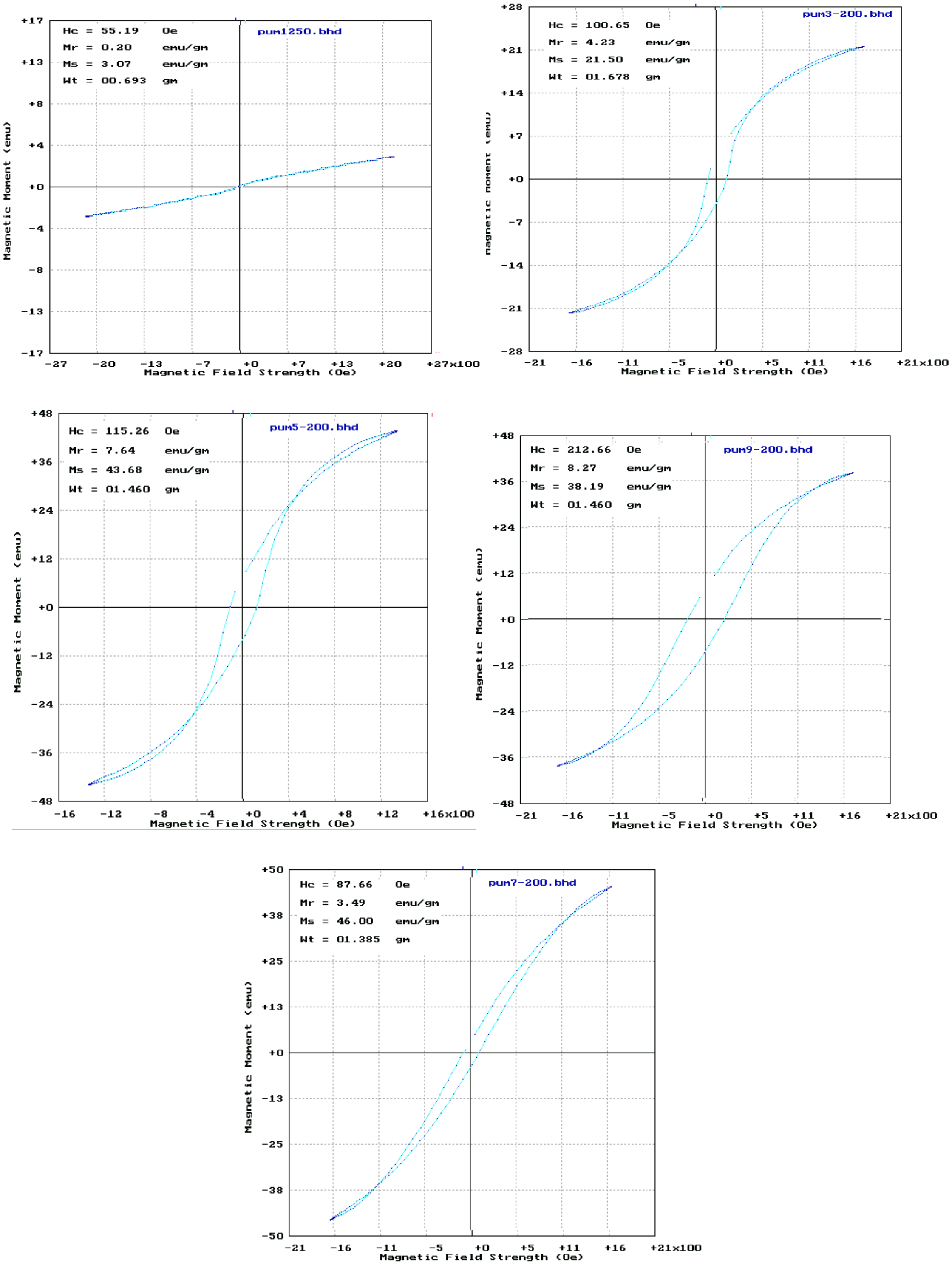

Fig. 4 Hysterisis plots of nanocrystalline $Ni_XZn_{1-X}Fe_2O_4$ ferrites

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Murthy et al [16] have studied the IR absorption in Ni-Zn ferrites. The vibrations in band positions are due to change in Fe^{3+}- O^{2-} bond distance in two crystal sites. Waldron [17] has attributed the band υ_1 around 600 cm^{-1} to the vibrations of tetrahedral complexes corresponding to highest restoring force causes stretching of Fe^{3+}- O^{2-} bonds where as bond υ_2 around 400 cm^{-1} is attributed to vibrations of octahedral complexes which are bond bending vibrations. The data listed in Table-1.

Fig. 4 shows magnetic hysteresis loops of the nanocrystalline Ni-Zn ferrites at room temperature. The values of saturation magnetization M_s, magnetic moment n_B and Y-K angles are also listed in table 1. The saturation magnetization increases with decrease in Zn content. It is maximum for Zn = 0.3 and then decreases. It is also observed that for all Ni-Zn nanoferrites the change of coercivity with Zn content is not consistant. The change in coercivity arises from physical pinning of magnetic domain walls likely due to grain boundries. Simillar results observed in thin films of Mn-Zn nanoferrites[18]. The Y-K angles for Zn = 0.1 is zero indicating Neel' two sublattice model can be applied to explain variation of magnetization of sample. As Zn content increases, 'A' sublattice magnetically diluted to reduce A-B exchange. Due to this 'B' spins no longer remain parallel but they can't as expressed by Yafet - Kittle [19].

CONCLUSION

Nanocrystalline $Ni_xZn_{1-x}Fe_2O_4$ ferrites with varying x were synthesized by sucrose process. XRD pattern confirms the synthesis of fully crystalline single phase Ni-Zn nanoferrites. TEM micrograph studies shows nanocrystalline nature of samples with average crystallite size between 30nm to50nm. The values of saturation magnetization and magnetic moment increases with decrease in Zn content up to Zn = 0.7 and then increases. The nonzero values of Y-K angles show triangular arrangement of spins on 'B' site leading to reduction in A-B interaction.

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