

Structural and magnetic characterizations of Co substituted Ni-Cu-Zn nanoferrites

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Co substituted Ni-Cu-Zn nanoferrites with the compositional formula $\text{Co}_x \text{Ni}_{0.5-x} \text{Cu}_{0.3} \text{Zn}_{0.2} \text{Fe}_2\text{O}_4$ (where $x=0.0, 0.1, 0.2$) were synthesized by Citrate Precursor method. The as prepared samples were calcined at 950°C for 30 min using a conventional muffle furnace. Characterizations were carried out using XRD, FESEM and VSM techniques. XRD peaks conform to spinel type structure. FESEM micrographs showed surface morphology. Magnetic characterizations were carried out by employing VSM technique. This paper investigates the synthesized samples as potential electronic materials for Multilayer Chip Inductor (MLCI).

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1. Introduction

Nanomaterials like spinel ferrites are soft magnetic materials and have gained lots of attention from scientific communities as they play a promising role in understanding the function of magnetic materials in various fields [1]. Spinel ferrites have many applications in the technology sector such as high-density magnetic storage, sensor, drug delivery for biomedical applications, computer circuitry and catalysts with high efficiency [2, 3]. It has been reported that magnetic properties can be enhanced by doping Co^{2+} ion in Ni-Zn ferrites [4]. Hence, in the present study, Co doped Ni-Cu-Zn nanoferrites with the compositional formula $\text{Co}_x \text{Ni}_{0.5-x} \text{Cu}_{0.3} \text{Zn}_{0.2} \text{Fe}_2\text{O}_4$ were synthesized and calcined at 950°C for 30 min. XRD, FESEM and VSM technique were employed for further investigation.

2. Experimental details

Raw materials used for the synthesis of nanocrystalline $\text{Co}_x \text{Ni}_{0.5-x} \text{Cu}_{0.3} \text{Zn}_{0.2} \text{Fe}_2\text{O}_4$ ferrite by sol gel auto combustion method or citrate precursor method are $(\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O})$ (98% purity, Merck, India), $(\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O})$ ($\geq 99\%$ purity, Merck, India), $(\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O})$ ($\geq 96\%$ purity, Merck, India), $(\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O})$ ($\geq 98\%$ purity, SRL,), $(\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O})$ (98% purity, Merck, India), $(\text{C}_6\text{H}_8\text{O}_7 \cdot \text{H}_2\text{O})$ ($\geq 99\%$ purity, Merck, India).

These chemicals serve as the precursors for the present investigation. Specific quantities of metal nitrates and citric acid are measured in the molar ratio of 1:1 and dissolved in double distilled water. The solution is then stirred for a few minutes to dissolve the solutes. Then, diluted ammonia solution is added drop-wise in the above chemical solution till the pH value becomes 7. The solution is then stirred for 30 minutes and temperature is increased to 100°C and stirring is continued until a viscous gel is formed. The gel ignited itself and formed loose ferrite powder. The product obtained was calcined at 950°C for 30 minutes in a muffle furnace [5].

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3. Results and discussions

3.1. XRD analysis

XRD pattern was recorded using Bruker D8 ADVANCE. Fig. 1. depicts the XRD spectra of Co substituted Ni-Cu-Zn nanoferrites with varying content of Co ($x=0.0, 0.1$ and 0.2). The diffraction peaks conform to the standard JCPDS card of reference code 01-071-850. In order to estimate the crystallite sizes, Debye-Scherrer's formula was used:

$$D = \frac{0.9\lambda}{\beta \cos \theta} \quad (1)$$

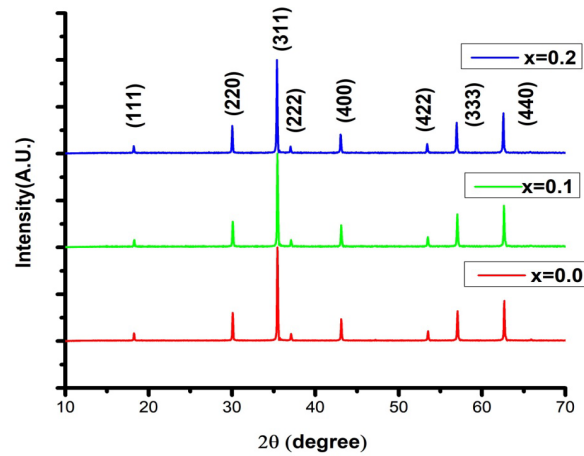


Fig. 1. XRD pattern of $\text{Co}_x \text{Ni}_{0.5-x} \text{Cu}_{0.3} \text{Zn}_{0.2} \text{Fe}_2\text{O}_4$.

Table 1. Crystallite size, Theoretical density and Lattice parameter of $\text{Co}_x \text{Ni}_{0.5-x} \text{Cu}_{0.3} \text{Zn}_{0.2} \text{Fe}_2\text{O}_4$.

x	Crystallite size(nm)	Theo. density (gm/cm ³)	Lattice constant(Å)
0.0	71.17	5.47	8.387
0.1	65.97	5.47	8.387
0.2	66.17	5.45	8.401

where, β is the FWHM of the peak (311), λ is the CuK_α wavelength of X-ray and θ is the diffracted angle. The crystallite size varies from 71.17 nm to 66.17 nm. The lattice constant was found out using the formula:

$$a = d(h^2 + k^2 + l^2)^{1/2} \quad (2)$$

where, h, k, l denote the Miller indices and d represents the interplanar spacing.

Theoretical density was found out using the following equation:

$$d_x = \frac{8M}{Na^3} \quad (3)$$

where, the symbols signify their usual meanings. These measured values from equations (1), (2) and (3) are presented in Table 1.

Decrease of crystallite size with cobalt doping from $x=0.0$ to $x=0.1$ can be explained with distribution effect of cations over A sites and B sites i.e. tetrahedral and octahedral sites respectively. When Co^{2+} with an ionic radii of 0.74 Å replaces Zn^{2+} with an ionic radii of 0.82 Å at

tetrahedral site, shrinkage of unit cell results. When the concentration of cobalt is raised from $x=0.1$ to $x=0.2$, the crystallite size increases [6].

Lattice constant increases with increase of Co^{2+} doping concentration as Co^{2+} has larger ionic radius of 0.74 \AA which replaces Ni^{2+} of ionic radius 0.69 \AA . This is in accordance with Vegard's law [7].

3.2. SEM analysis

FESEM micrographs were recorded using Zeiss Sigma. Fig. 2 depicts the surface morphology of $\text{Co}_x\text{Ni}_{0.5-x}\text{Cu}_{0.3}\text{Zn}_{0.2}\text{Fe}_2\text{O}_4$ nanoparticles. Agglomeration of particles is observed in these micrographs.

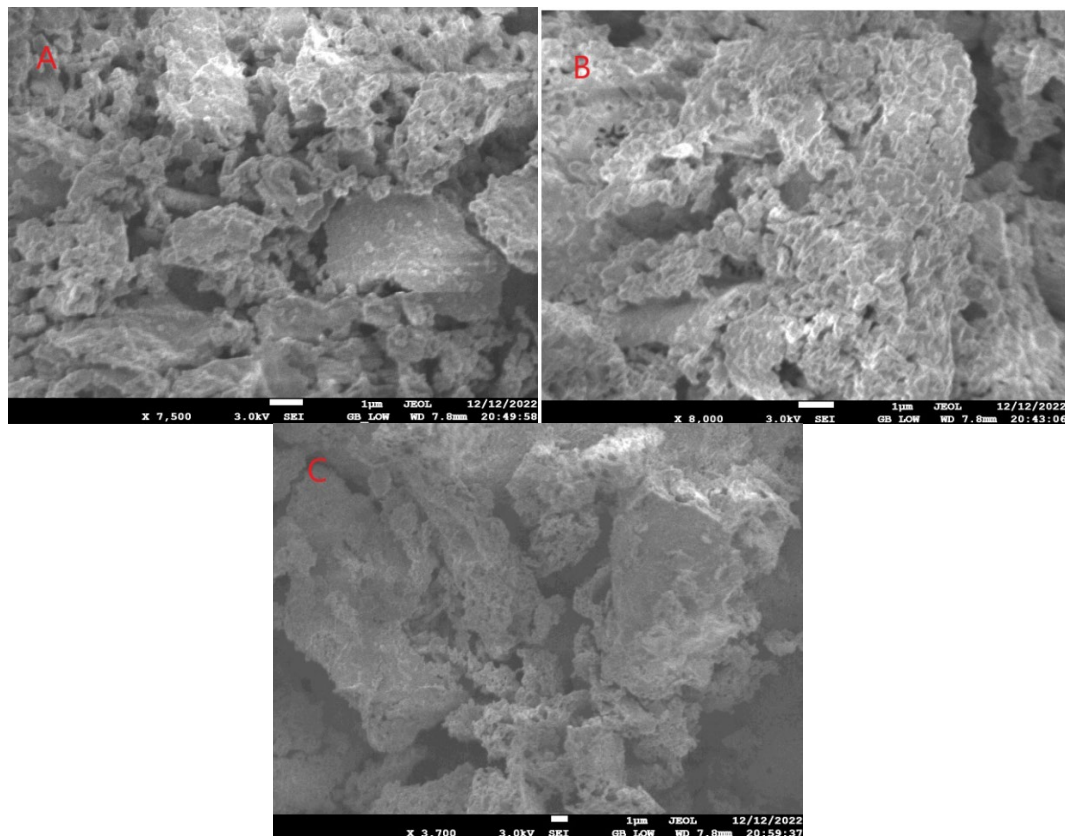


Fig. 2. FESEM image of $\text{Co}_x\text{Ni}_{0.5-x}\text{Cu}_{0.3}\text{Zn}_{0.2}\text{Fe}_2\text{O}_4$ ($A x=0.0, B x=0.1, C x=0.2$).

3.3. Magnetic characterization

The magnetic parameters were measured by employing VSM Lakeshore 7410 series. Fig.3 shows the M-H curve for VSM study at room temperature for $\text{Co}_x\text{Ni}_{0.5-x}\text{Cu}_{0.3}\text{Zn}_{0.2}\text{Fe}_2\text{O}_4$ nanoferrites. The values of H_{ci} , M_s and M_r so obtained are shown in Table 2.

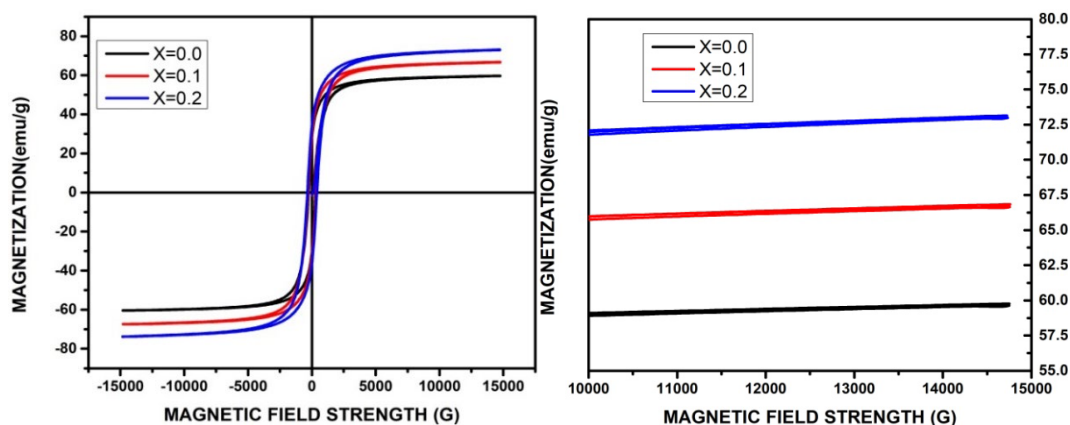


Fig. 3. M - H curve for $\text{Co}_x \text{Ni}_{0.5-x} \text{Cu}_{0.3} \text{Zn}_{0.2} \text{Fe}_2\text{O}_4$

Table 2. H_{ci} , M_s and M_r of $\text{Co}_x \text{Ni}_{0.5-x} \text{Cu}_{0.3} \text{Zn}_{0.2} \text{Fe}_2\text{O}_4$.

X	Coercivity (H_{ci})(G)	Magnetization (M_s)(emu/g)	Retentivity (M_r)(emu/g)
0.0	275.37	60.087	28.788
0.1	324.15	67.139	31.340
0.2	374.54	73.466	35.432

The increase in the value of M_s with Co^{2+} doping can be explained by the presence of different cations of varying magnetic moments at the A and B sub lattices. Co^{2+} ions of higher magnetic moment ($3\mu_B$) replaces the Ni^{2+} ions of lower magnetic moment ($2\mu_B$) resulting in the increase of net magnetic moment with Co^{2+} incorporation. This consequently increases the saturation magnetization (M_s) with the increase in Co^{2+} content [8].

4. Conclusion

$\text{Co}_x \text{Ni}_{0.5-x} \text{Cu}_{0.3} \text{Zn}_{0.2} \text{Fe}_2\text{O}_4$ nanoferrites were effectively synthesized by Citrate Precursor method. The confirmation of spinel type structure formation was obtained from the analysis of XRD. The crystallite size lies in the nanometer range. It was observed that lattice constant increases with increasing Co^{2+} content. Further, the net magnetization increased with Co^{2+} doping. These materials will serve as potential materials for MLCIs and magnetic recorders due to the suitable calcination temperatures and moderate magnetization values.

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