Effect of Al3+ Substitution On Magnetic and DC Electrical Resistivity Properties of NiZnCu Nanoferrites

N MURALI (✉ muraliphdau@gmail.com)
Andhra University  https://orcid.org/0000-0002-8272-2802

Research Article

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Effect of Al$^{3+}$ substitution on magnetic and DC electrical resistivity properties of NiZnCu nanoferrites

G. Vishnu Priya¹, S. Ravi Kumar¹, B. Aruna¹, D. Vijaya Lakshmi¹, T. Gunavardhana Naidu², P. Geetha³, M. K. Raju⁴, D. Parajuli⁵, N. Murali⁶,* P. V. L. Narayana¹,*

¹Department of Nuclear Physics, Andhra University, Visakhapatnam – 530003, A. P., India
²Department of BS&H, Aditya Institute of Technology & Management, Tekkali, Srikakulam
³Department of BS&H, GMR Institute of Technology, Rajam, Srikakulam – 532127, A. P.
⁴Department Physics, WISTM Engineering College, Pinagadi, Visakhapatnam, A.P., India
⁵Department of Physics, Andhra University, Visakhapatnam – 530003, A. P.
⁶Department of Engineering Physics, AUCE (A), Andhra University, Visakhapatnam

*Corresponding email: muraliphdau@gmail.com, dr.pvln@andhrauniversity.edu.in

Abstract: Al substituted Ni$_{0.4}$Zn$_{0.35}$Cu$_{0.25}$Fe$_{2-x}$Al$_{x}$O$_4$ (x = 0.00, 0.05, 0.10, 0.15, 0.20) samples is synthesized using the sol-gel auto-combustion process. X-ray diffraction shows its cubic spinel structure. The lattice constant decreases as the Al$^{3+}$ content increases. The sizes of the crystallites are also decreasing in the range of 32.15 nm to 22.89 nm. The wavenumbers of tetrahedral and octahedral sites sighted in the FT-IR spectra are similar to that of the precursor. The increment in the Al$^{3+}$ content increases the DC conductivity. The electrical resistivity decrease with an increase in the temperature, i.e., it has a negative temperature coefficient with resistance similar to semiconductors. VSM results show their isotropic nature forming single domain ferrimagnetic particles. The resultant material is widely significant, as indicated by its result.

Keywords: Al$^{3+}$ substituted NiZnCu ferrite, XRD, FT-IR, Magnetic, Electrical properties.

1. Introduction

These spinel nanomaterials or ferrites with cations in their tetrahedral and octahedral sites are used as soft magnetic materials in everyday life [1-3]. Changing their internal with doping can make them applicable in more advanced devices. Most of the spinel ferrite materials have M$^{2+}$Fe$_2$$^{3+}$O$_4^2$ the type of structure where M is a Transitional Metal Cation. M is on the tetrahedral site, and Fe on the octahedral site of the spinel. This soft magnetic ferrite’s high resistivity and low power loss made them be used in multilayered chip inductors, microwave absorption materials, information storage systems, transformer core,
computer circuitry, and electronic communication. Among them, Ni-Zn ferrite is highly stable at a higher frequency [4, 5].

Ni–Cu–Zn ferrites are used in Multilayer Chip Inductors (MLCI) and Surface Mounting Devices (SMDs). As a result, they are utilized in cellular phones, video cameras, notebook computers, personal wireless communication systems, etc. their properties can be changed by varying the concentration of Cu$^{2+}$ and found to be more applicable in inductive devices like transformers. The ferrites prepared at low temperatures are used in multilayer power inductors and transformers [6-8].

The purpose of this study is to a sol-gel auto-combustion method for the synthesis of the samples Ni$_{0.5}$Zn$_{0.5-x}$Cu$_x$Fe$_2$O$_4$ (0.0 ≤ x ≤ 0.4) nanoparticles and investigates the influence of chemical composition, DC electrical resistivity, and magnetic properties. Ni–Cu–Zn ferrites have been studied for different electrical and magnetic applications [9]. Albeit high dielectric constants, i.e., electrical properties, have been reported for these ferrites, the correspondingly high dielectric loss, especially at high operating frequencies, hampers their application in reality [10].

A spinel system's magnetic parameters collectively respond to the synthesis method, cation types, and occupancy in different sites. The distribution of cations depends on the types of bonding and the ionic radii of the cations. A. Dzunuzovic et al. [11] fabricated Zn-substituted Ni-ferrites and studied their structural and morphological properties. The results show that Zn-substituted Ni-ferrites are of cubic spinel structure and the grain size of samples increases with Zn composition. Charalampos Stergiou [12] reported dielectric and magnetic properties of rare-earth (Y and La) substituted Ni-Co-Zn spinel ferrites. The dielectric constant is increased with the rare-earth substitution due to the improvement of the dielectric orientation polarization. D.R.S. Gangaswamy et al. [13] fabricated Co-substituted Ni–Zn–Mg ferrite and investigated the magnetic properties. It is found that saturation magnetization is decreased, and the soft magnetic nature increases with Co substitution. S.J. Haralkar et al. [14] prepared Cr-substituted Mg-Zn ferrite. They studied the cation distribution. It is observed that the fraction of Fe ions in octahedral sites decreases, and the fraction of Cr ions in octahedral sites increases with the substitution of Cr. Yu Gao et al. [15] synthesized Li-substituted Ni–Zn ferrite and investigated elastic, structural, and magnetic properties. They observed that the characteristic band of ferrite shifts towards a lower frequency with Li [16].

Different ion substituted ferrites have been reported in the literature; very few are given above, but Ni–Zn–Cu–Al is still not fabricated and studied.
In this research, we have reported that the synthesis of Ni-Zn-Cu ferrites by the sol-gel auto combustion method by doping a portion of nonmagnetic Al$^{3+}$ content and studying the concentration of contents on the structure dc electrical resistivity and magnetic properties.

2. Experimental techniques

Aluminium Substituted Nickel-Zinc-Copper nanoparticles are prepared by the sol-gel auto-combustion method. 99.99% pure Nickel nitrate, Zinc nitrate, Copper nitrate, Aluminium nitrate, Iron nitrate, and citric acid monohydrate with the molecular formula (Ni(NO$_3$)$_2$·6H$_2$O), (Zn(NO$_3$)$_2$·6H$_2$O), Cu(NO$_3$)$_3$·6H$_2$O, Al(NO$_3$)$_3$·9H$_2$O, (Fe(NO$_3$)$_3$·9H$_2$O) and (C$_6$H$_8$O$_7$·H$_2$O) respectively, as the starting materials. They are mixed in such a ratio that Ni$_{0.4}$Zn$_{0.35}$Cu$_{0.25}$Fe$_{2-x}$Al$_x$O$_4$ (x = 0, 0.05, 0.1, 0.15, 0.2) samples are prepared. These metal nitrates and citric acid were mixed in a molar ratio of 1:1 and were dissolved in the distilled water to get a clear solution. The solution was made neutral by adding liquid ammonia. The solution was then stirred in a magnetic stirrer maintained at 100 °C for 4 h, decanted, and dried at normal temperature for 40 h. The flakes thus obtained were combusted and converted into powder. The powder was sintered in a muffle furnace at 800 °C for 4 h at 5°/min.

We used Rigaku X-ray diffractometer (Rigaku Miniflex II) incorporated with CuKα radiation of wavelength = 1.5406 Å for the structural property, TESCAN, MIRA II LMH SEM with attached Inca Oxford EDX for textural and compositional images, FT-IR analysis for the detection of functional group, EZ VSM model for the magnetic at room temperature. Few drops of polyvinyl alcohol were mixed with the powder for shaping them into disc-like pallets after pressing them in a die under the hydraulic press of 5 tons. The pallets were then made as electrodes by sintering them in 800°C in a muffle furnace and polishing their flat sides with gold. Their conductivity was checked by the two-probe DC resistivity method.

3. Results and discussions

3.1 XRD Studies

The XRD plots of different Ni$_{0.4}$Zn$_{0.35}$Cu$_{0.25}$Fe$_{2-x}$Al$_x$O$_4$ (x = 0, 0.05, 0.1, 0.15, 0.2) samples are shown in Figure 1. The structure of the sample is found to be a cubic spinel structure according to the JCPDS card No.48-0489. The lattice constant ‘a’ is determined with the following relation [17].

\[ a = d_{hkl} \sqrt{h^2 + k^2 + l^2} \]
where $d_{hkl}$ is more interplane spacing for given hkl planes and is calculated by Bragg’s law, the highest intensity (311) peak indicates the crystallite's appropriate orientation to measure its degree crystalline nature to find the average crystallite size of all samples. Debye-Scherer’s formula gives the average size of the crystallite size [18].

$$D_{311} = \frac{0.9\lambda}{\beta \cos \theta}$$

where $D_{311}$, $\lambda$, $\beta$ and $\theta$ are volume-averaged crystallite size, the wavelength of X-ray (1.5406 Å), full width at half maximum of (311) peak and diffraction angle respectively.
Figure 1: X-ray diffraction patterns of Al-doped NiZnCu nanoferrites
TABLE 1: Lattice parameters of Al-doped NiZnCu nanoferrites

| Concentration (x) | a (Å) | Crystallite (nm) | Space Group |
|-------------------|-------|------------------|-------------|
| 0.0               | 8.343 | 32.15            | Fd-3m       |
| 0.05              | 8.339 | 28.14            | Fd-3m       |
| 0.1               | 8.331 | 26.48            | Fd-3m       |
| 0.15              | 8.328 | 24.65            | Fd-3m       |
| 0.2               | 8.323 | 22.89            | Fd-3m       |

Figure 2: Variation of lattice constant and crystallite size of Al-doped NiZnCu nanoferrites

The crystallite size and lattice parameters of the composition values are listed in Table 1. The crystallite size first decreases from 32.15 nm to 22.89 nm (for x = 0.0 to 0.2). In contrast, the lattice parameter decreases with increases in the Al$^{3+}$ ions concentration. This is due to the greater ionic radius of Fe$^{3+}$ ions (0.67 Å) [19] as compared to Al$^{3+}$ (0.51 Å) [20], thereby expanding the unit cell or decreasing the lattice constant [21], as shown in figure 3.
The obtained value of the lattice parameter of the base sample is 8.343 Å to 8.323 Å. It is well-matched with the value reported in the literature [22]. When the smaller Al3+ ions replaced the larger Fe3+ ions, the unit cell shrank while preserving the overall cubic symmetry. The lattice compression may also be due to the partial oxidation of Fe3+, Al3+. The variation of the lattice constant is more significant in the smaller size of the nanoparticle.

The diffraction peak width (β) is inversely proportional to the crystallite size from Scherer's formula. The increase in the lattice parameter expands the volume of the unit cell accordingly. Sintering decreases the lattice defects and involved strain but facilitates the crystals' coalition increasing in particle size.

3.2 Field-Effect Scanning Electron Microscope (FESEM) with EDS

The FESEM images from Figure 3 show that Ni0.4Zn0.35Cu0.25Fe2-xAlxO4(x = 0, 0.05, 0.1, 0.15, 0.2) are not miscible, and the average size of the grain ranges from 0.2 to 0.4 μm. The grains sizes are nearly equal [23]. The microstructure images like grain size, pores, inclusions, grain boundaries, particle size, homogeneity, defects, etc., can be obtained with the Electron microscope's help. The smaller grain size with low porosity controls excessive spin-wave production, which is essential for microwave devices. Similarly, the large grain size supports the mobility of the domain wall, resulting in high permeability with low coercive value. Also, the eddy current losses are checked by the grain boundaries acting as current barriers.
3.3 Fourier Transformed Infrared (FTIR) Spectroscopy studies

The FTIR spectroscopy helps determine the different functional groups for their wavenumbers or how the absorption occurs. Our sample's Ni$_{0.4}$Zn$_{0.35}$Cu$_{0.25}$Fe$_{2-x}$Al$_x$O$_4$ ($x = 0, 0.05, 0.1, 0.15, 0.2$) spectra within the range of 400-1200 cm$^{-1}$ are shown in figure 5. The spinel or inverse spinel ferrite can show four IR active regions or high intensity or non-zero dipole moment regions in the vibrational spectra. First, three of them are the resultant of tetrahedral and octahedral compounds, and the fourth is due to the lattice vibration of the tetrahedral cation. The two absorption bands at wavenumbers 580.4 to 598.84 cm$^{-1}$ and 402.1 to 405.35 cm$^{-1}$ are seen as two depressions in figure 6, and values listed in Table 2 are the two respective characteristics bands of each spinel ferrite. The bond length between Fe$^{3+}$ and O$^{2-}$ is varied for the sample composition, resulting in the deviation in the peak position of $v_1$ and $v_2$ towards the high-frequency region [24].
Figure 5: Infrared spectra of Al-doped NiZnCu nanoferrites

Table 2: Absorption bands of Al-doped NiZnCu nanoferrites

| Concentration (x) | Tetrahedral $\nu_1$(cm$^{-1}$) | Octahedral $\nu_2$(cm$^{-1}$) |
|-------------------|--------------------------------|-------------------------------|
| 0.0               | 580.40                         | 402.1                         |
| 0.05              | 582.85                         | 403.48                        |
| 0.1               | 585.92                         | 404.46                        |
| 0.15              | 591.05                         | 407.85                        |
| 0.2               | 598.84                         | 405.35                        |
3.4 Magnetic properties

Ferrites have antimagnetic moments with unequal magnitudes. As a result, they have a large value of spontaneous magnetization. The exchange integral, depending on interatomic distance, is negative for ferrite. This indirect exchange interaction through oxygen ions limits the easy flow of electrons. So, ferrites have high resistivity [25].

Figure 6: Hysteresis curve of Al-doped NiZnCu nanoferrites

The magnetic properties like saturation permeability, coercivity, susceptibility, Curie temperature, etc., of ferrites, depend on the concentration of metal ions on both the octahedral and tetrahedral sites. The hysteresis loop shapes, resistivity, ac conductivity, and dielectric constant depend on the ferrite structure. So, these properties are more sensitive to the system. These properties can be changed by adding external magnetic or non-magnetic metal ions. The hysteresis curves of our respective samples are as shown in figure 6. Figure 7 shows the values of coercivity (H_c), saturation magnetization (M_s), etc., that are important for their magnetic properties. The values of M_s and H_c are listed in Table 3 [26].
Table 3: Ms and Hc of Al-doped NiZnCu nanoferrites

| Concentration (x) | Ms (emu/g) | Hc (Oe) |
|-------------------|------------|---------|
| 0.0               | 14.1       | 325     |
| 0.05              | 39.2       | 285     |
| 0.1               | 44.5       | 264     |
| 0.15              | 43.1       | 166     |
| 0.2               | 25.2       | 959     |

Figure 7: Variation of saturation magnetization and coercivity of Al-doped NiZnCu nanoferrites

From table 3, Ms values are decreases in decreasing Al$^{3+}$ concentration. Adding Al$^{3+}$ ions to the Ni-Zn-Cu mixture, they exchange few magnetic ions Fe$^{3+}$ and Ni$^{2+}$ in B- site increases AB interaction that interrupts the antiparallel spin B site resulting from the increase in total magnetization [27]. A similar phenomenon occurs on the A site. According to Weiss's Molecular field theory, the A-B and B-A interaction dominates the A-A and B-B interaction resulting in the hysteresis loop [28-30].
3.5 DC Electrical Resistivity

The DC resistivities of Ni$_{0.4}$Zn$_{0.35}$Cu$_{0.25}$Fe$_{2-x}$Al$_x$O$_4$ (x = 0, 0.05, 0.1, 0.15, 0.2) ferrites nanoparticles initially decreases rapidly, then steadily and then continue with the further increase in Al$^{3+}$ concentration. The plot of DC resistivity vs. temperature for the Al$^{3+}$ substituted Ni-Zn-Cu is shown in figure 8. A graph between log $\rho$ and 1000/T is a straight line. It shows that the resistivity decreases with an increase in temperature showing semiconducting behaviour. The slope of the line gives the activation energies of the ferrite samples. The dc resistivity data are used in Arrhenius to find the thermally activated charge carriers' activation energy [31].

![Figure 8: Temperature dependence of DC resistivity of Al-doped NiZnCu nanoferrites](image)

The Arrhenius relation is,

$$\rho = \rho_o e^{-\frac{\Delta E}{RT}}$$

where $\rho$ is the dc electrical resistivity at temperature T, $\rho_o$ is the pre-exponential factor, $\Delta E$ is the activation energy, $K$ is the Boltzmann constant, and T is the absolute temperature. The
calculated values of activation energies of the synthesized ferrite nanoparticles samples are shown in Figure 9. Verwey and de Boer hopping mechanism helps to interpret the resistivity variation for the Al-doped Ni-Zn-Cu ferrite nanoparticles. Electron hopping occurs between ions of the same element located at different valance states and the two sites. During sintering of the ferrites, the divalent and trivalent iron ions can be produced and exist in octahedral sites that help in electrical conduction through $\text{Fe}^{2+} \leftrightarrow \text{Fe}^{3+}$ hopping mechanism. If the ferrite's sintering temperature is higher, more $\text{Fe}^{2+}$ ions are produced, thereby accelerating the hopping process. The hopping process is possible in $\text{Fe}^{2+} \leftrightarrow \text{Fe}^{3+}$ and $\text{Al}^{3+} \leftrightarrow \text{Al}^{2+}$ existing together in a system [32].

Figure 9: Activation energies of the Al-doped NiZnCu nanoferrites

About the above calculation, as in figure 8, the activation energies are found to be in the order of 0.48 to 0.60 eV which is for the $\text{Fe}^{2+} \leftrightarrow \text{Fe}^{3+}$ electron hopping mechanism. It indicates that the major conduction mechanism is the $\text{Fe}^{2+} \leftrightarrow \text{Fe}^{3+}$ process. Besides, the conduction processes such as $\text{Fe}^{2+} + \text{Zn}^{3+} \leftrightarrow \text{Fe}^{3+} + \text{Zn}^{2+}$ ions require relatively more energy for electron hopping, so the energy required could be slightly more than 0.48 eV. The temperature-dependent resistivity and associated activation energies indicate the compositional dependence of resistivity [33].
4. CONCLUSIONS

Sol-gel auto-combustion method is utilized to fabricate nanocrystalline Ni$_{0.4}$Zn$_{0.35}$Cu$_{0.25}$Fe$_{2-x}$Al$_x$O$_4$($x = 0$, $0.05$, $0.1$, $0.15$, $0.2$) ferrite NPs. The structure of the ferrite shown by the X-ray diffraction is single phase cubic spinel. The decreasing crystallite size values increasing the Al concentration. In comparison, the lattice parameter decreases with the increases in Al$^{3+}$ ions concentration. This is due to the greater ionic radius of Fe$^{3+}$ ions (0.67 Å) than Al$^{3+}$ (0.51 Å), thereby expanding the unit cell or decreasing the lattice constant. FESEM reveals microstructural growth along with heat action. The FTIR spectrum exhibits a prominent attribute of ferrite microstructure and a major impact on ingredients' mixture. The magnetic measurements show that magnetization reduces and coercivity enhances. DC resistivity is decreasing with an increase in the content of copper due to its highly conducting property. The electrical resistivity decrease with an increase in the temperature, i.e., it has a negative temperature coefficient with resistance similar to semiconductors. The temperature-dependent resistivity and associated activation energies indicate the compositional dependence of resistivity.
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