



Structural, magnetic and electrical properties of Al³⁺ substituted Ni–Zn ferrite nanoparticles

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ABSTRACT

Nanocrystalline ferrite materials having the general formula Ni_{0.7}Zn_{0.3}Fe_{2-x}Al_xO₄ (0.0 ≤ x ≤ 0.5) have been synthesized by citrate-gel auto combustion method and characterized using X-ray diffraction (XRD), energy dispersive X-ray (EDX), field emission scanning electron microscopy (FE-SEM), dc magnetization, dielectric and impedance spectroscopy measurements. XRD studies confirm that all the samples show single phase cubic spinel structure. The crystallite size of Ni_{0.7}Zn_{0.3}Fe_{2-x}Al_xO₄ (0.0 ≤ x ≤ 0.5) nanoparticles calculated using the Debye–Scherrer formula was found in the range of 13–17 nm. The value of lattice parameter ‘a’ is found to decrease with increasing Al³⁺ content. EDX patterns confirm the compositional formation of the synthesized samples. FE-SEM micrographs show that all the samples have nano-crystalline behavior and particles show spherical shape. The variation of dielectric properties ε', ε'', and tan δ with frequency shows the dispersion behavior which is explained in the light of Maxwell–Wagner type of interfacial polarization in accordance with the Koop's phenomenological theory. The dc magnetization studies infer that magnetic moment of Ni_{0.7}Zn_{0.3}Fe_{2-x}Al_xO₄ (0.0 ≤ x ≤ 0.5) nanoparticles was found to decrease with Al doping. Impedance spectroscopy techniques have been used to investigate the effect of grain and grain boundary on the electrical properties of the synthesized compounds.

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1. Introduction

During the last few decades, ferrites have achieved a primary position of economic and engineering importance within the family of magnetic materials because of their excellent physical properties. Practically all TV sets have ferrite cores in the fly back transformers, while portable radios make use of a pencil of ferrite as an antenna core. Long distance carrier telephone circuits are employing ferrite cores in high quality filter coils and transformers. Various physical properties of nanocrystalline ferrites are highly influenced by distribution of cations among the sub lattices, nature of grain (shape, size, and orientation), grain boundaries, voids, in homogeneities, surface layers, contacts, etc. [1–3]. The information about the associated physical parameters of the microstructural components is important; since, the overall property of the materials is determined by these components. Progresses in the use and development of new ferrites have been rapid as compared to other areas of research [4–9]. One important characteristic of

ferrites is their high values of resistivity and low eddy current losses [10–13], which make them ideal for high frequency applications. For microwave applications, the dielectric properties such as dielectric constant and dielectric loss are very important as the dielectric constant affects the thickness of microwave absorbing layer and the dielectric loss factor (tan δ) of a material determines dissipation of the electrical energy. This dissipation may be due to electrical conduction, dielectric relaxation, dielectric resonance and loss from non-linear processes. Recently, Kumar et al. have reported that ferrites also behave like multiferroics [14]. The major interest in the synthesis of uniform magnetic nanocrystals with controllable size and their physical properties has stimulated a search for general schemes to prepare high-quality magnetic nanocrystals of varying compositions. There are many researchers who studied the magnetic properties of nickel–zinc ferrites but for the nonmagnetic properties such as electrical conductivity and dielectric properties are seldom reported.

In this work, chemical-route technique has been chosen for the synthesis of Ni_{0.7}Zn_{0.3}Fe_{2-x}Al_xO₄ (0.0 ≤ x ≤ 0.5) nanoparticles. Various techniques such as; XRD, FE-SEM, dc magnetization, dielectric and impedance spectroscopy measurements have been used to study their structural, magnetic and electrical properties.

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Structural studies infer that all the samples have single phase cubic spinel structure and lattice parameters decreases with increase in Al content. The saturation magnetization calculated from magnetic hysteresis loop was found to decrease with increase in Al doping. FE-SEM micrographs show the nanocrystalline behavior of the samples. Evaluation of electrical conductivity reveals a wealth of information as regards the usefulness of these materials for various applications. Moreover the study of dielectric sheds light on the behavior of charge carriers under the ac field, their mobility and the mechanism of conduction.

2. Experimental

Nanoparticles of $\text{Ni}_{0.7}\text{Zn}_{0.3}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$ ($0.0 \leq x \leq 0.5$) ferrite were prepared by using citrate gel auto combustion method. The chemical reagents used in this work were $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ and $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ as starting materials. All the chemicals were of analytical grade. We use citric acid $\text{C}(\text{OH})(\text{COOH})(\text{CH}_2\text{-COOH})_2 \cdot \text{H}_2\text{O}$ (M.W.210.14) in this method because citric acid is a weak acid and has three carboxylic and one hydroxyl group for coordinating metal ions and therefore enhances the homogeneous mixing. Citric acid helps for the homogenous distribution and segregation of the metal ions. During water dehydration, it suppresses the precipitation of metal nitrates because it has electronegative oxygen atoms interacting with electropositive metal ions. Therefore, at a relative low temperature the precursors can form a homogenous single phase. The ammonia is used to adjust the pH for improving the complication, gel formation and also to improve the solubility of metal ions. Metal nitrates taken in the required stoichiometric ratio were dissolved in a minimum amount of distilled water and mixed together. The mixed metal nitrate solution was then added to the citric acid solution in 1:1 molar ratio. The pH value of the clear solution thus obtained was unity. Analytical grade liquor ammonia (30%) was then added drop-wise under constant stirring in order to reach the pH of the solution up to 6. The resulting solution was continuously heated on the magnetic stirrer at 70°C in order to allow gel formation. The gel so formed is kept in an air oven at 100°C for 36 h in order to remove the adsorbed water [15]. During this process the gel swells into a fluffy mass, which eventually breaks into brittle flakes. This precursor powder was then calcined at 600°C for 8 h to obtain final product. The rate of heating and cooling was maintained $10^\circ/\text{min}$. The resultant powders were grind into fine particles using an agate mortar and pestle. The structural characterization of all samples was carried out by Rigaku X-ray diffractometer (Rigaku Miniflex II) using the $\text{CuK}\alpha$ radiation (wavelength $\lambda = 1.5406 \text{ \AA}$). Field emission scanning electron microscopy (FE-SEM) images were obtained using a TESCAN, MIRA II LMH microscope. The composition was determined by energy dispersive X-ray spectroscopy (EDX, Inca Oxford, attached to the FE-SEM). For the FE-SEM and EDX measurements, the nanoparticles of $\text{Ni}_{0.7}\text{Zn}_{0.3}\text{Fe}_{2-x}\text{Al}_x\text{O}_4$ ($0.0 \leq x \leq 0.5$) ferrite were dispersed homogeneously in ethanol using ultrasonic treatment. A minute drop of nanoparticles solution was cast on to a glass slide followed by subsequently drying in air before transfer it into the microscope. The dielectric and impedance spectroscopy measurements were carried out using LCR HI-Tester (HIOKI3532-50) at room temperature in the frequency range from 42 Hz to 5 MHz. Shielded test leads were used for the electrical connections from the LCR meter to the sample in order to avoid any parasitic impedance. The samples were made in the form of circular pellets. Before starting the measurements the samples were heated at 100°C for 1 h, so as to homogenize the charge carriers and to remove the moisture content therein. The surface of the disks was coated on adjacent faces with silver paste, thereby forming parallel plate capacitor geometry.

The value of real dielectric constant (ϵ') has been calculated using the following relation:

$$\epsilon' = \frac{c_p \times t}{\epsilon_0 A} \quad (1)$$

where $\epsilon_0 = 8.85 \times 10^{-14} \text{ F/cm}$, known as permittivity of the free space, t is the thickness of pellets, A is the cross sectional area of the flat surface of the pellets and c_p is the capacitance of the pellet in (F). The imaginary dielectric constant has been calculated by the relation:

$$\epsilon'' = \epsilon' \tan \delta \quad (2)$$

where $\tan \delta$ is the dielectric loss tangent which is proportional to the 'loss' of energy from the applied field into the sample (in fact this energy is dissipated into heat) and therefore denoted as dielectric loss.

The loss tangent has been calculated from the following relation:

$$\tan \delta = \frac{1}{2\pi f \epsilon_0 \epsilon' \rho} \quad (3)$$

here f is the frequency of the applied field and ρ is the resistivity. The magnetic measurements were performed using Quantum design physical properties measurement system (PPMS 6000).

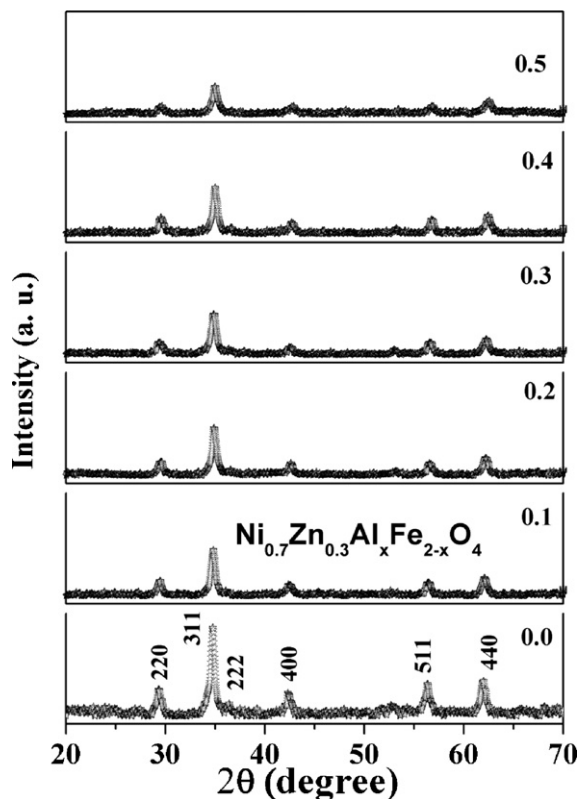


Fig. 1. X-ray diffraction pattern of $\text{Ni}_{0.7}\text{Zn}_{0.3}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$ ($0.0 \leq x \leq 0.5$) ferrite nanoparticles.

3. Results and discussion

Fig. 1 highlights the XRD patterns of $\text{Ni}_{0.7}\text{Zn}_{0.3}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$ ($0.0 \leq x \leq 0.5$) ferrite nanoparticles. It can be clearly seen from Fig. 1 that all samples exhibit single phase cubic spinel structure and exclude presence of any undesirable secondary phase. Moreover, the broadening of the XRD peaks indicate that $\text{Ni}_{0.7}\text{Zn}_{0.3}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$ ($0.0 \leq x \leq 0.5$) ferrite have nano-crystalline behavior. The crystallite size $\text{Ni}_{0.7}\text{Zn}_{0.3}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$ ($0.0 \leq x \leq 0.5$) ferrite nanoparticles was calculated from the most intense peak (3 1 1) of XRD data using Debye Scherrer formalism:

$$t = \frac{0.9\lambda}{\beta \cos \theta} \quad (4)$$

where $\beta = (\beta_M^2 - \beta_I^2)^{1/2}$. Here λ is X-ray wavelength (1.54 \AA for $\text{Cu K}\alpha$), β_M and β_I are the measured and instrumental broadening in radians respectively and θ is the Bragg's angle in degrees. The calculated value of particle size was found to increase from 13 to 17 nm (see Table 1) with Al doping, which implies that Al doping favor the particle growth. The lattice parameters calculated using Powder-X software was found to decrease from 8.516 \AA to 8.385 \AA (see Table 1) with increase in Al^{3+} content. The values of the lattice parameters observed in $\text{Ni}_{0.7}\text{Zn}_{0.3}\text{Al}_x\text{Fe}_{2-x}\text{O}_4$ ($0.0 \leq x \leq 0.5$) ferrite nanoparticles are in good agreement with the reported value for cubic spinel ferrites [16,17]. The decrease in lattice parameters with increase in Al^{3+} doping can be explained on the basis of difference in the ionic radii of Fe and Al ions, since, the ionic radius of Al^{3+} ion (0.57 \AA) is smaller than that of Fe^{3+} ion (0.67 \AA). The variation in the X-ray density (theoretical density) and apparent density (experimental density) with Al^{3+} ion concentration is shown in Table 1. It is clear from the table that the X-ray density decreases with the increase of Al^{3+} ion concentrations. This can be explained on the basis of the fact that the density and the atomic weight of the Al atoms

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