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Structural Analysis of the Indium Substituted Ni-Cu-Zn Ferrites using XRD Technique

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ABSTRACT

Nickel-copper–zinc ferrite samples, $Ni_{0.35}Cu_{0.05}Zn_{0.60}In_xFe_{2-x}O_4$, with x = 0.00 to x =0.125 were prepared by sol-gel auto combustion method. The chemical phase analysis has been carried out by X-ray powder diffraction (XRD) method, which confirms the formation single phased cubic spinel structure with a crystallite size ranging from 25 to 45 nm. While increasing indium concentration, the lattice constant increases. On the XRD plot, this causes the diffraction peaks to shift toward lower 2θ angles. Since indium has a much higher atomic weight than iron, the X-ray density calculated from the XRD data will increase significantly as indium replaces iron. A higher peak intensity for indium-doped samples usually suggests improved crystallinity.

1. Introduction

Ferrite materials of spinel structural formula AB_2O_4 are mainly composed of about 70% iron oxide (Fe_2O_3) and about 30% of other metal oxides including MgO, MnO, NiO, CuO, etc., [1]. Each spinel unit cell contains eight formula units. In each unit cell, there are 64 tetrahedral sites (A sites) and 32 octahedral sites (B sites) [2]. On the basis of distribution of metal ions at (A) and (B) site, spinel ferrites are of three types; namely normal, inverse and random spinel ferrite. The high electrical resistivity, low eddy current and dielectric loss, high saturation magnetization, high permeability, good chemical stability, ease of preparation, low cost of production etc., are the remarkable features of spinel ferrites. Magnetic spinel nano ferrites have attracted the interest of the researchers due to their importance in understanding the fundamentals of nano magnetism and their wide range of applications. They found potential applications in bio- medicine such as magnetic resonance imaging (MRI), targeted drug delivery, magnetic separation [3]. NiCuZn ferrites are excellent soft magnetic materials in high frequency device due to their low cost, high resistivity and low eddy current losses that have been extensively studied for multi-layer chip inductor (MLCI) applications [4]. Extensive research has investigated how various dopants influence the Ni-Cu-Zn ferrite matrix, specifically aiming to improve its dielectric, magnetic, and structural parameters for advanced applications. Over the past few decades, research has pivoted toward developing innovative techniques to enhance the structural and magnetic characteristics of soft magnetic spinel ferrites [5]. Specifically, Ni-Cu-Zn ferrites have garnered significant attention due to their critical role in the fabrication of multilayer chip inductors (MLCIs). While various synthesis strategies—ranging from traditional solid-state ceramic methods to sol-gel self-propagation—have been employed, the sol-gel auto-combustion route stands out [6]. This method is increasingly preferred for its ability to produce highly stoichiometric, nano-scaled powders at reduced processing temperatures and lower operational costs [7].

This present work focus on the synthesis of NiCuZn ferrite powders by a solgel auto-combustion technique. This is a novel way with a unique combination of the chemical solgel process and the combustion process. The process has the advantages of inexpensive precursors, a simple preparation method, and a resulting nano-sized, homogeneous, highly reactive powder [8]. Also, the work is discussing about X-ray powder diffraction (XRD) method, formation of single phased cubic spinel structure, crystallite size, the lattice constant and X-ray density.

2. Experimental Methods

For the synthesis of nanocrystalline $Ni_{0.35}Cu_{0.05}Zn_{0.60}In_xFe_{2-x}O_4$ferrites (where x = 0.0 to 0.125 in increments of 0.025) high-purity (99.9%) precursors were utilized via the citrate-nitrate auto-combustion technique. The metal nitrates specifically nickel nitrate ($Ni(NO_3).6H_2O$), copper nitrate ($Cu(NO_3).6H_2O$), zinc nitrate ($Zn(NO_3).6H_2O$), indium nitrate ($In(NO_3).9H_2O$), and ferric nitrate ($Fe(NO_3).9H_2O$) served as the primary reactants, while citric acid monohydrate $C_6H_8O_7.H_2O$ acted as the fuel and chelating agent.

Aqueous solutions of metal nitrates and citric acid were combined, maintaining a stoichiometric 1:3 molar ratio. The resulting mixture was subjected to continuous agitation via a magnetic stirrer to ensure the formation of a uniform citrate-nitrate precursor solution [9]. The precursor solution was evaporated through heating until a gel phase was established. This gel was subsequently dehydrated in an air oven at 100 °C to eliminate residual moisture. During dehydration, the material expanded into a voluminous, fluffy mass before fragmenting into brittle flakes. The resulting precursor was calcined at 600 °C for 4 h to yield the nanocrystalline ferrites. A comprehensive schematic of the synthesis and characterization workflow is presented in Fig. 1.

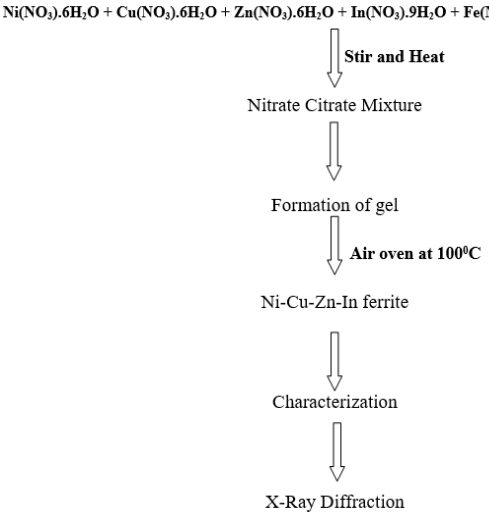

Fig. 1 Flowchart for the synthesis and characterization of Ni–Cu–Zn–In Nano ferrites

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Phase identification and the verification of chemical reaction completion were conducted using X-ray diffraction (XRD). Patterns were recorded on a Rigaku D/MAX-IIA diffractometer utilizing CuKα radiation. Data collection was performed over a 2θ range of 20–70° with a constant scanning rate of 2.9×10^{-4} rad/s. The lattice parameters and X-ray densities of the samples were calculated from the diffraction pattern (Fig. 1) [10]. The sintered density was determined from the mass and bulk volume of the samples. By comparing the values of both densities, the porosity for each sample was determined.

3. Results and Discussion

Fig. 2 illustrates the XRD pattern of developed indium substituted NiCuZn ferrite samples with x = 0.00 – 0.125 for the general composition $Ni_{0.35}Cu_{0.05}Zn_{0.60}In_xFe_{2-x}O_4$. The XRD spectra showed the crystallite state of ferrites that possess cubic spinel structure. The obtained planes (220), (311), (222), (400), (422), (511) and (440) confirmed the formation of cubic spinel structure having Fd3m space group. No any further impurity peaks are obtained in the XRD, which clearly specifies the developed material is of good quality in terms of purity [11].

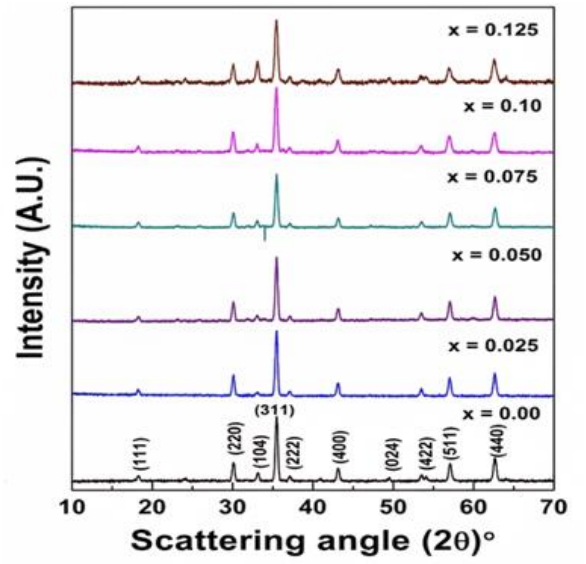

Fig. 2 XRD pattern of developed indium substituted NiCuZn ferrite samples

Solgel auto combustion method is a bottom-up chemical route the resulting powder is nanocrystalline the XRD report will typically include, crystalline size (D) calculated using the Scherrer formula, typically ranges from 25 nm to 45 nm. The X-ray density (ρ_x) usually increases because the atomic weight of indium (114.82 u) is much higher than that of iron (55.84 u) [12]. Effect of indium on lattice parameters as Indium In^{3+} has a larger iconic radius 0.80 Å than Iron (Fe^{3+}) 0.645 Å as a result the XRD report will show, peak shifts towards lower 2θ angles as indium concentration increases, there is linear increase in lattice parameter, the cell volume also increases [13].

4. Conclusion

This study demonstrates that the sol-gel auto-combustion technique is an effective route for producing high-purity In^{3+} substituted NiCuZn ferrites. Structural characterization via XRD verified that all samples maintained a cubic spinel phase without impurity formation. As the substitution of Indium ions was achieved without disrupting the lattice integrity, the increase in Indium concentration x factors the formation of NiCuZn ferrites with higher density. X-ray density depends on the lattice constant and molecular weight of the samples shows an increasing trend with increasing Indium content. These nano-ferrites show significant potential for miniaturized magnetic components. The high homogeneity and refined particle size achieved at a relatively low calcination temperature (600 °C) highlight the advantages of this processing approach.

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