

Cr³⁺ substitution effect on Co-Cu and Cu-Co nano ferrites on structural and morphological properties

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Abstract

The Cr³⁺ substituted Co-Cu (Co_{0.7}Cu_{0.3}Fe_{2-x}Cr_xO₄) and Cu-Co (Cu_{0.7}Co_{0.3}Fe_{2-x}Cr_xO₄) where $x = 0.0, 0.05, 0.1, 0.15, 0.2$ and 0.25 nanoferrite composite were prepared with the sol-gel approach. Their structural, DC electrical resistivity and magnetic properties were analyzed. XRD shows the single-phase spinel ferrite. Adding Cr³⁺ ions decreases the lattice volume and the size of the crystallite respectively. FESEM images show non-spherical particles on a largely uniform surface shape with decreasing grain size on doping Cr³⁺. The FTIR pattern supports the XRD patterns for spinel ferrite.

Keywords

Nanoferrites, Cr³⁺ substitute, XRD, FESEM, FTIR.

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1 Introduction

The investigation of nano-scale magnetic materials and their properties is currently a highly active area of research due to their unique and captivating attributes. These properties often differ from those of larger bulk substances due to their high surface-to-volume ratio [1–3]. Nanoscale particles, with applications in diverse fields such as bio-processing, color imaging, memory storage devices, ferrofluids, and magnetic refrigeration, have gained significant attention [4–6]. Ferrites can be categorized as either soft or hard based on their mag-

netization and demagnetization characteristics [7]. These ferrites encompass four crystal chemistry categories: spinel ferrites, garnet ferrites, magnetoplumbite ferrites, and orthorhombic ferrites [8–10]. Among these, spinel ferrites, denoted by the chemical formula MFe₂O₄ where M represents a divalent metal cation, have emerged as the most extensively researched due to their remarkable optical and magnetic properties [11–13]. Spinel ferrites find applications in sensors, electromagnets, optoelectronic components, high-frequency devices, and more [14, 15]. They exhibit exceptional attributes such as higher resistivity values, magneti-

zation, permeability, and lower eddy current losses [16]. The distinct properties of spinel ferrites can be influenced by factors such as sintering temperature, preparation methods, choice of dopant ions, particle size, and agglomeration [17–19]. Extensive studies have focused on utilizing divalent and trivalent metal ions to enhance the electrical, optical, and magnetic characteristics of ferrites. Notable substitutions, like replacing Fe^{3+} with Cr^{3+} in cobalt ferrite, have been explored without altering the spinel structure [20]. While there has been considerable exploration of divalent ion replacements, trivalent cations have not been as extensively studied as potential substitutes in spinel materials [21].

The most notable cobalt and spinel ferrite compound is CoFe_2O_4 , which possesses a combination of advantageous features suitable for high-density magnetic recording applications. These include low cost, high coercivity, mechanical hardness, and chemical stability, making it an ideal choice [22]. Within this structure, half of the Fe^{3+} ions occupy tetrahedral sites, while the remaining half occupies octahedral sites, resulting in an inverse spinel configuration [23, 24]. By substituting different metal ions into the cobalt lattice, ferrite materials with novel properties are generated. The characteristics of these materials are significantly influenced by factors such as preparation conditions, the quantity and type of substituent [17, 25–28].

When non-magnetic copper is introduced as a substitution into the cobalt ferrite lattice, it is expected to induce distortions in the spinel structure [29–32]. The addition of copper results in an intriguing distribution of cations across the interstitial sites A and B within the cobalt ferrite lattice [33]. Cu^{2+} is a Jahn-Teller ion with degenerate orbitals, exhibiting high electrical conductivity in its ground state [34]. The introduction of Cu^{2+} ions can induce crystal distortions [35]. Reports suggest that the positioning of Cu^{2+} ions at A and B sites influences the extent of crystal distortion they cause [36–38].

Our recent research has investigated the impact of Cr^{3+} substitution in cobalt-copper ferrite compositions ($\text{Co}_{0.7}\text{Cu}_{0.3}\text{Fe}_{2-x}\text{Cr}_x\text{O}_4$) and ($\text{Cu}_{0.7}\text{Co}_{0.3}\text{Fe}_{2-x}\text{Cr}_x\text{O}_4$) [2, 39–42]. It was observed that the inclusion of Cr^{3+} ions in the cobalt ferrite lattice leads to a decrease in saturation magnetization due to the lower magnetic moment of Cr^{3+} ions compared to Fe^{3+} ions. Additionally, the electrical resistivity increased as the concentration of Cr^{3+} ions rose. This phenomenon can be attributed to the single stable oxidation state of chromium ions, which reduces the efficiency of conduction involving Fe^{2+} and Fe^{3+} ions, resulting in higher resistivity. Various techniques have been employed to produce cobalt-copper ferrite nanoparticles [43, 44]. Among these, the sol-gel process, known for gener-

ating heterogeneous and crystalline nanoparticles, stands out as a chemical method capable of effectively producing ferrite nanoparticles [45]. This process involves hydrolyzing and condensing metal precursors to form a three-dimensional inorganic system. Metal precursors are utilized in this low-temperature synthesis process [46]. This study aims to investigate the structural and morphological properties of Co-Cu ($\text{Cu}_{0.7}\text{Co}_{0.3}\text{Fe}_{2-x}\text{Cr}_x\text{O}_4$) and Cu-Co ($\text{Co}_{0.7}\text{Cu}_{0.3}\text{Fe}_{2-x}\text{Cr}_x\text{O}_4$) nanoferrites at varying levels of x , specifically $x = 0.0, 0.05, 0.1, 0.15,$ and 0.2 . The synthesis of the samples will involve utilizing the sol-gel auto-combustion method. This analysis will employ techniques such as X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM), and Fourier-transform infrared spectroscopy (FT-IR) to examine the resulting structures and morphologies.

2 Experimental Techniques

The initial precursors for the synthesized materials include cobalt, copper, iron, chromium nitrates, and citric acid, all obtained from Sigma Aldrich with a purity of 98%. A chelating agent, citric acid, was used to form complexes with the metal nitrates. To create the citric acid solution, the nitrates were mixed with citric acid in a 1:1 ratio. The resulting mixture was stirred magnetically at temperatures between 80 and 90^o Celsius for a duration of 10-12 hours, leading to the formation of a viscous gel. This gel was then subjected to drying at 1000 degrees Celsius for 6 hours. The product was decomposed using spontaneous self-ignition and subsequently powdered using a mortar and pestle. The powdered material was annealed at 1100^oC for 4 hours to eliminate impurities.

Phase identification of the Cr^{3+} substituted Co-Cu ($\text{Co}_{0.7}\text{Cu}_{0.3}\text{Fe}_{2-x}\text{Cr}_x\text{O}_4$) and Cu-Co ($\text{Cu}_{0.7}\text{Co}_{0.3}\text{Fe}_{2-x}\text{Cr}_x\text{O}_4$) nanoferrites was carried out using X-ray diffraction (XRD) with a Cu-K x-ray radiation source. X'PERT PRO software was employed to analyze the X-ray diffraction patterns within the range of 20 to 80^o diffraction angles. The surface morphology and elemental distribution of the synthesized samples were studied using FESEM (Field Emission Scanning Electron Microscopy) models Quanta 250 and FEI D9393. Furthermore, different vibrational modes in the prepared samples were determined using FTIR (Fourier-Transform Infrared) spectrometers, 400 FTIR, and FIR (Far-Infrared).

Force constants calculations

$$v_1^2 = \frac{1}{2\pi c} \sqrt{\frac{K}{\mu}} \quad (1)$$

where $\mu = \frac{\mu_1\mu_2}{\mu_1 + \mu_2}$ for two atoms systems. Tetra-

hedral and Octahedral Force constants K_T and K_O are directly proportional to the vibration frequency and molecular weights (μ_A, μ_B) by the following relations as suggested by Waldron [47]:

$$K_T = 4\pi^2 c^2 \mu_A v_1^2 \quad (2)$$

$$K_O = 4\pi^2 c^2 \mu_B v_2^2 \quad (3)$$

Cation distribution data was used to estimate the molecular weights of A- and B-sites. The molecular weights (μ_A and μ_B) and force constants (K_T and K_O) are directly proportional to each other.

The values of tetrahedral and octahedral radii (r_A and r_B) can be calculated using the equations given below:

$$r_A = \text{Cobalt content} \times r_{Co} + \text{Copper content} \times r_{Cu} + \text{Iron content} \times r_{Fe}$$

and $r_B =$

$$\frac{1}{2} [\text{Cromium content} \times r_{Cr} + \text{Iron content} \times r_{Fe}]$$

The theoretical lattice constants have also been estimated using the following formulae by proposing the cationic distribution of metal ions in the spinel lattice.

$$r_A = (u - \frac{1}{4})a_{th}\sqrt{3} - R_o \quad (4)$$

$$r_B = (\frac{5}{8} - u)a_{th} - R_o \quad (5)$$

$$a_{th} = \frac{8}{3\sqrt{3}} [(r_A + R_o) + \sqrt{3}(r_A + R_o)] \quad (6)$$

Where, r_A and r_B are radii of tetrahedral and octahedral sites respectively. a_{th} is theoretical lattice constants. R_o is the radius of the oxygen ion. u is the oxygen positional parameter. For the fcc structure, R_o is 0.375. In the Co-Cu ferrite system, the radii of Cr^{3+} and Co^{2+} ions are greater than the space occupied by the oxygen ions. This will cause distortion in the cubic lattice and therefore the oxygen parameter may differ from its usual value of 3/8 [48].

3 Results and Discussion

3.1 XRD Study

The X-ray diffraction (XRD) patterns of Cr^{3+} substituted Co-Cu ($Co_{0.7}Cu_{0.3}Fe_{2-x}Cr_xO_4$) and Cu-Co ($Cu_{0.7}Co_{0.3}Fe_{2-x}Cr_xO_4$) nanoferrites, where $x = 0.0, 0.05, 0.1, 0.15, 0.2,$ and $0.25,$ are presented in Figure 1 (a) and (b). These patterns reveal the presence of a single-phase spinel structure [?,35,49], specifically related to the Fd3m space group with a card number of 22-1086 [50]. The XRD patterns exhibit distinct and well-defined peaks in the (111), (220), (311), (222), (400), (422), (511), (440), (620), and (533) planes, indicating a high degree of crystallinity that aligns closely with existing literature [28,51,52].

The values in Table 1 exhibit a gradual decrease due to the substitution of Cr^{3+} ions. For Co-Cu nanoferrites, the lattice parameter reduces from 8.4498 Å to 8.4321 Å, while for Cu-Co nanoferrites, it decreases from 8.4441 Å to 8.4099 Å. This decrease is attributed to the larger ionic radii of Fe^{3+} ions (0.645) compared to Cr^{3+} ions (0.615) [53].

3.2 FESEM analysis

Utilizing field effect scanning electron microscopy (FESEM), the characterization of shape and grain size in Cr^{3+} substituted Co-Cu ($Co_{0.7}Cu_{0.3}Fe_{2-x}Cr_xO_4$) and Cu-Co ($Cu_{0.7}Co_{0.3}Fe_{2-x}Cr_xO_4$) nanoferrites, where $x = 0.0, 0.05, 0.1, 0.15, 0.2,$ and $0.25,$ has been conducted. The FESEM micrographs of the synthesized nanoferrites are depicted in Figures 3 (a) and (b). Extensive research has been undertaken by various investigators to gain a deeper understanding of fine-grain morphology [54,55]. The FESEM micrographs reveal agglomerated, spherical, refined, and densely packed magnetic nanoparticles with an average grain size ranging from 50 to 100 nm. Remarkably, this study corroborates the findings by establishing a robust correlation between the grain size deduced from the observed FESEM micrograph analysis and the crystallite size determined from the XRD diffraction patterns of the synthesized samples [53].

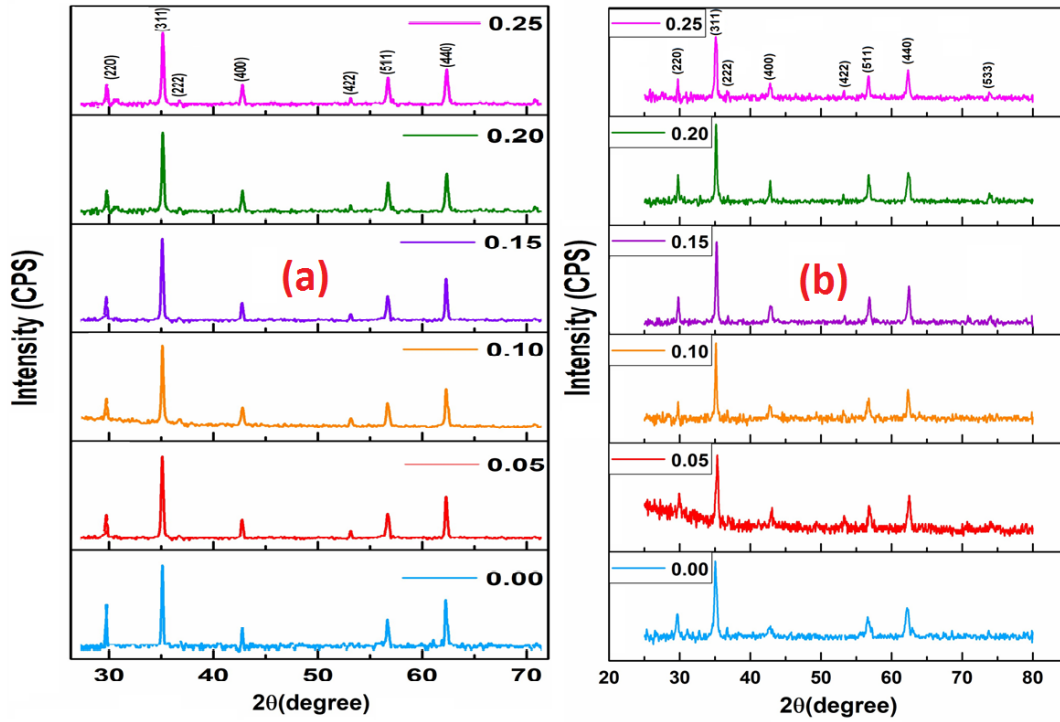


Figure 1: (a) and (b): XRD images of Cr^{3+} doped Co-Cu and Cu-Co nano ferrite.

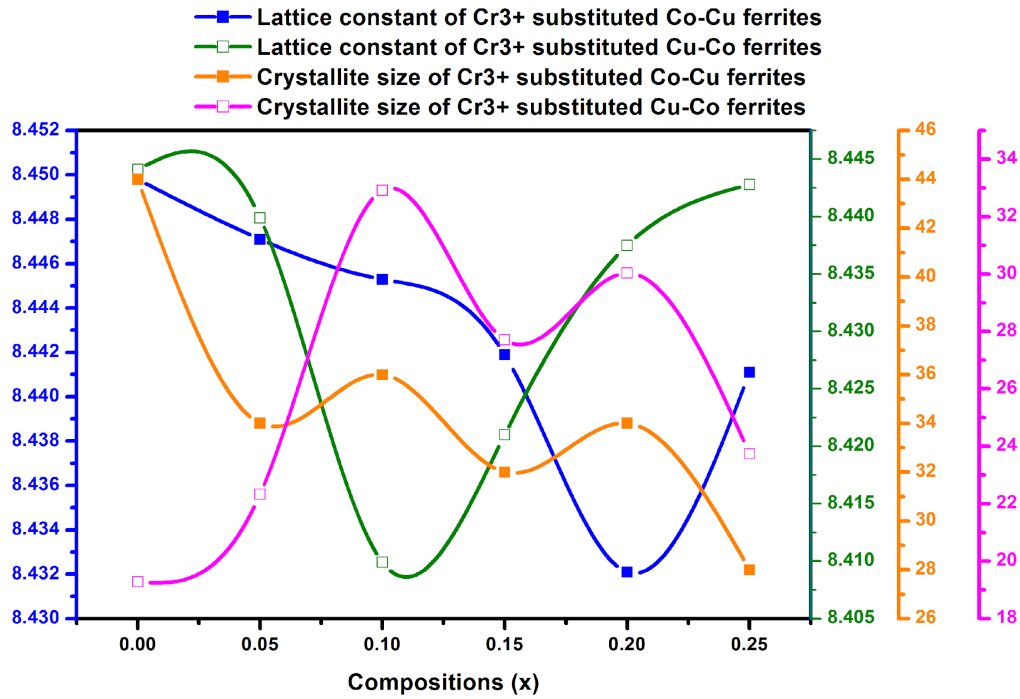
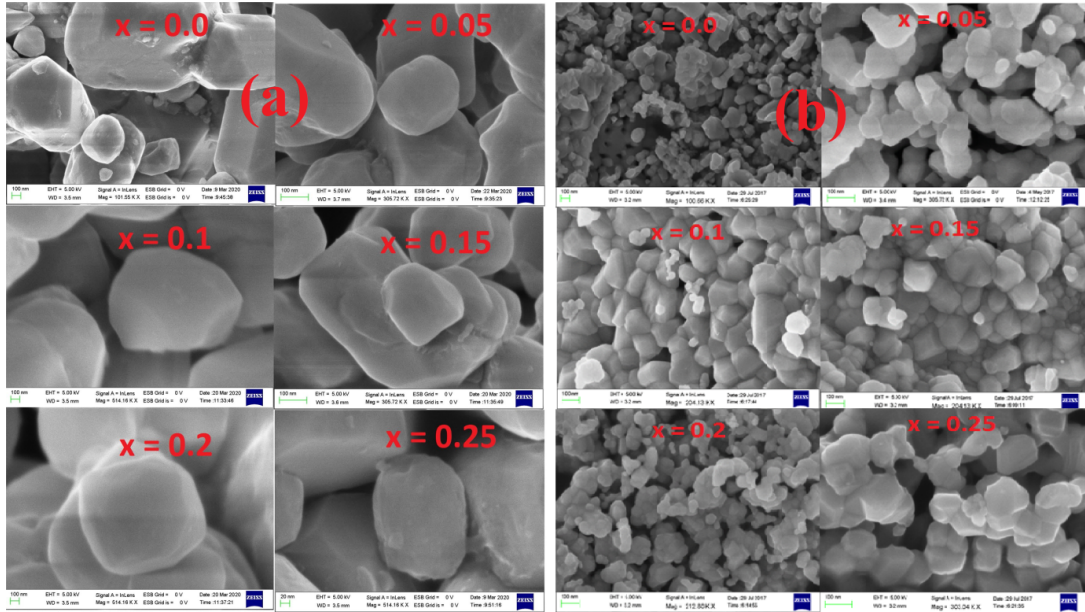


Figure 2: Lattice constant and crystallite size vs. composition of the Cr^{3+} substituted Co-Cu and Cu-Co nano ferrite.

Table 1: Experimental values for different concentrations of the samples

Composition (x)	Cr ³⁺ substituted Co-Cu		Cr ³⁺ substituted Cu-Co	
	Lattice Constant (Å)	Crystallite size (nm)	Lattice Constant (Å)	Crystallite size (nm)
x = 0.0	8.4498	44	8.4441	19.28
x = 0.05	8.4471	34	8.4399	22.33
x = 0.1	8.4453	36	8.4099	32.92
x = 0.15	8.4419	32	8.4210	27.71
x = 0.2	8.4321	34	8.4375	30.04
x = 0.25	8.4411	28	8.4428	23.74

Figure 3: (a) and (b): FESEM images of Cr³⁺ substituted Co-Cu and Cu-Co nanoferrite.

The substitution flexibility within available lattice sites diminishes as particle growth occurs, particularly when the chosen substituted element exhibits a pronounced affinity for a specific site. This results in a more controlled nucleation process and particle size. It is widely recognized that cobalt and chromium ions exhibit a strong preference for occupying octahedral sites within the lattice structure [56].

3.3 FTIR analysis

FTIR spectroscopy is employed to analyze vibrational modes, investigate structural stability, and assess cation distribution within interstitial sites. Figures 4 (a) and (b) display the typical FTIR spectra of Cr³⁺ substituted Co-Cu (Co_{0.7}Cu_{0.3}Fe_{2-x}Cr_xO₄) and Cu-Co (Cu_{0.7}Co_{0.3}Fe_{2-x}Cr_xO₄) nano ferrites, where x = 0.0, 0.05, 0.1, 0.15, 0.2, and 0.25. These spectra were obtained using the samples prepared by the

sol-gel method, with samples sintered at 1100^oC for 4 hours. The transmittance spectra were executed within wavenumbers of 350 to 800 cm⁻¹ at room temperature. Notably, below 800 cm⁻¹, the presence of two prominent absorption bands affirms the formation of a spinel structure in the synthesized ferrites [57]. Furthermore, these spectra offer insights into the functional groups present in the ferrite compositions. Two distinct vibrational bands, labeled v₁ and v₂, arise due to stretching vibrations occurring at both octahedral and tetrahedral sites. The vibrational band v₁ corresponds to the stretching vibrations of M³⁺-O₂ (M³⁺ = Cr³⁺ and Fe³⁺) bonds in octahedral sites. In contrast, the vibrational band v₂ can be attributed to the intrinsic vibrations of M²⁺-O₂ (M²⁺ = Co²⁺, Cu²⁺, and Fe²⁺) bonds in tetrahedral sites. This distinction arises due to the shorter Fe-O bond length (0.189 nm) in tetrahedral sites compared to octahedral sites (0.199 nm). Consequently, all ferrites exhibit these

two distinct bands, influenced by the closer coupling of Fe^{3+} ions at A sites rather than B sites. The positions of these vibrational bands alter as the Cr^{3+} doping density increases. This phenomenon is due to the substitution of larger Fe^{3+} ions by smaller Cr^{3+} ions, resulting in a change in site radius. A notable relationship between site radius and fundamental frequency is observed, where a decrease in

site radius leads to an increase in the fundamental frequency [18]. The presence of substituted Cr^{3+} ions disrupts the ($\text{Fe}^{3+} - \text{O}_2$) connections and perturbs the lattice structure, leading to a broadening of the high-frequency absorption band [58]. Table 2 provides the A and B band positions for samples at different levels of Cr^{3+} doping.

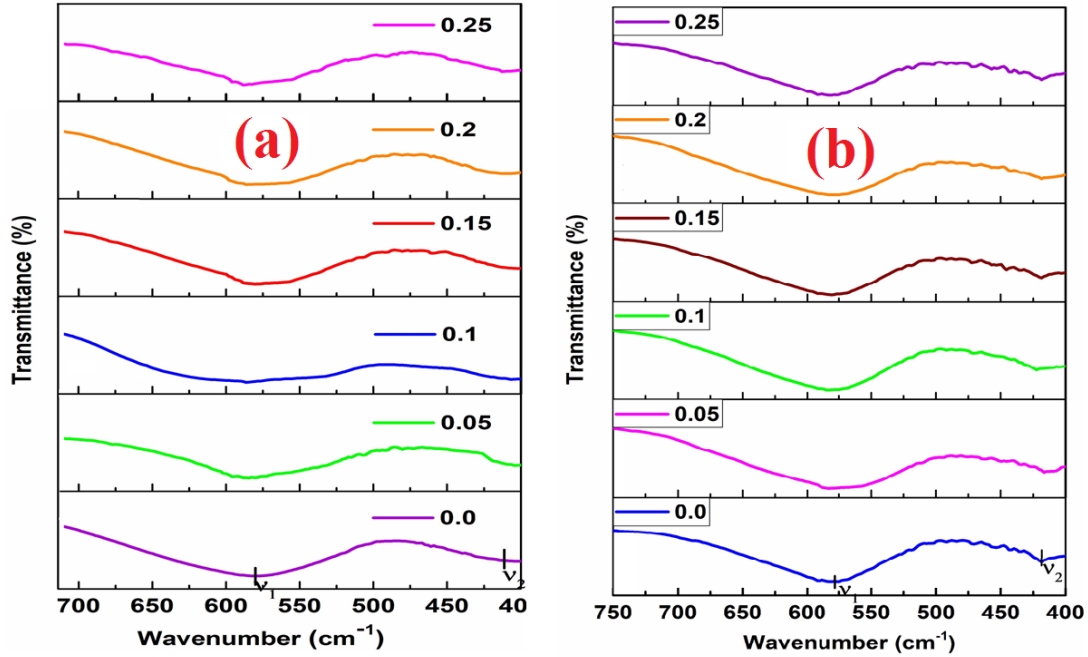


Figure 4: (a) and (b): FTIR spectra of Cr^{3+} -substituted Co-Cu and Cu-Co nanoferrite.

Table 2: Tetrahedral (ν_1) and Octahedral (ν_2) absorption bands $\text{Fe}^{3+} - \text{O}^{2-}$ bond force constants and of Cr^{3+} substituted Co-Cu and Cu-Co nanoferrite

Ferrite composition	Cr^{3+} substituted Co-Cu				Cr^{3+} substituted Cu-Co			
	ν_1 (cm^{-1})	ν_2 (cm^{-1})	$\text{Fe}^{3+} - \text{O}^{2-}$ bond force constant (10^5 dyne/cm)		ν_1 (cm^{-1})	ν_2 (cm^{-1})	$\text{Fe}^{3+} - \text{O}^{2-}$ bond force constant (10^5 dyne/cm)	
			K_T	K_O			K_T	K_O
$x = 0.0$	580.573	402.122	2.5791	1.3614	580.574	418.553	2.5701	1.3604
$x = 0.05$	584.431	404.051	2.5292	1.3675	582.502	420.624	2.6292	1.3675
$x = 0.1$	586.360	406.980	2.4717	1.3733	584.431	422.411	2.4717	1.3703
$x = 0.15$	582.502	402.122	2.4799	1.3796	580.164	418.239	2.4709	1.3706
$x = 0.2$	586.360	410.837	2.4554	1.3980	580.844	418.695	2.4604	1.3800
$x = 0.25$	588.288	412.766	2.4472	1.4041	580.175	418.508	2.4462	1.4031

4 Conclusion

The cost-effective sol-gel auto-combustion technique was successfully utilized to synthesize single-phase, well-crystallized ultrafine crystals of

Cr^{3+} substituted Co-Cu ($\text{Co}_{0.7}\text{Cu}_{0.3}\text{Fe}_{2-x}\text{Cr}_x\text{O}_4$) and Cu-Co ($\text{Cu}_{0.7}\text{Co}_{0.3}\text{Fe}_{2-x}\text{Cr}_x\text{O}_4$) nanoferrites, where $x = 0.0, 0.05, 0.1, 0.15, 0.2,$ and 0.25 . The resulting ferrite samples exhibit a face-centered cubic structure characterized by the Fd-3m space group.

The crystallite size of Cr^{3+} substituted Co-Cu is larger than that for Cu-Co. Notably, the cation distribution analysis indicates a preference for octahedral B-sites for Co^{2+} , Cu^{2+} , and Cr^{3+} ions within the lattice. The force constant is directly proportional to the atomic weights of the constituents in the respective lattice sites. The data reveals that the Cu-Co ferrite substituted with Cr is slightly more efficient than its counterpart.

Data Declaration

Data can be obtained from the corresponding author on request.

Competing Interest

The authors declare they have no competing interests.

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