To Facile the Study of Structural, Morphological Properties of Novel, Synthesised Nano Particles of Indium Doped Ni-Co Ferrite

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ABSTRACT

Sol-gel technique is used for synthesis of Ni_{0.5}Co_{0.5}In_{x}Fe_{2-x}O_{4} (x=0.0,0.10,0.075,0.050,0.025) characterized by utilizing. The pre-arranged example sintering temperature is affirmed by TGA. The pre-arranged examples Sintering at 600°C at 4 hours. The Single-phase cubic spinel was affirmed by utilizing the X-beam diffraction (XRD) examination. The underlying boundary like cross lattice constant, porosity, x-beam thickness, hopping length and so on, were estimated by utilizing the XRD information. The lattice constant (a) expanded from 8.347 to 8.378 Å. The x-beam thickness 5.385 to 5.518 g/cm³ expanded. The floor morphology affirmed through examining SEM. The cation conveyance proposes that Co^{2+}, Ni^{2+}, In^{3+} show a mobility towards the octahedral-B site, Fe^{3+} debris are arbitrarily circulated at the tetrahedral-A and octahedral-B site. The Fourier transform Infrared Spectra (FTIR) of the Ni-Co ferrite framework have been broke down the ingestion power tops both tetrahedral (υ₁) and octahedral (υ₂) increments with expanding the In^{3+} particles.

Keywords: Ni-Co ferrite, Indium, Sol-gel method, SEM, Cation distribution.

INTRODUCTION

Nanocrystalline spinel ferrites having general compound equation MFe_{2}O_{4} (where, M is divalent particle from the 3d series like Ni, Mn, Zn, Cu, and Co) are extremely fascinating attractive materials1,2. Spinel ferrites are widely concentrated because of the wild scope of utilizations like inductors, transformers, high-recurrence gadgets, microwave gadgets, multi-facet chip inductors, information stockpiling, and telecom3-5. The spinel ferrite is sorted into three distinct sorts Normal spinel, inverse spinel, and random spinel. The inverse spinel is intriguing because of its magneto crystalline like and high saturation magnetization.6 Ni-Co ferrite7-12. Cobalt-Nickel ferrite shows the inverse spinel structure in which Fe^{3+} and Co^{3+} particles live at both octahedral and tetrahedral destinations while Ni^{3+} particles live just at the octahedral site13-14. Cobalt and nickel, the two ferrites, have a place with the classification of
inverse spinel ferrites. Fe$^{3+}$ particles supplanted by In$^{3+}$ particles change the grid boundary changed. Because of the relatively larger ionic span of In$^{3+}$ particles (0.8 Å) concerning 3d progress metal particles, indium particles catch octahedral voids and furthermore make create lattice distortions a few analysts have given an account of nonmagnetic In$^{3+}$ particles doped ferrite like Co$^{18,19}$, Cu$^{20,21}$ Ni$^{22}$ Ni-Zn$^{23}$ Ni-Co$^{24}$ Ni-Cu$_{10}$ Ni-Cu-Zn$^{25}$. Chandra et al.,$^{26}$ Concentrated on the Ni$_{0.5}$Co$_{0.5}$Fe$_2$O$_4$ nanoparticle utilizing two unique techniques and its attractive conduct of nanoparticles. K. M. Srinivasa Murthy et al.,$^{27}$ concentrated on the Union of Ce$^{3+}$ doped Co-Ni ferrites and study the primary, microstructural, and dielectric properties of auto applications. R M Rosnan et al.,$^{28}$ were researched the expansion of Mg replacement Co$_{0.5}$Ni$_{0.5}$Mg$_{3-x}$Fe$_2$O$_4$ nanoparticle its practices change in underly and attractive properties of ferrites. M. Junaid, et al.,$^{10}$ considered, Effect of indium replacement Cu$_{0.5}$Ni$_{0.5}$Fe$_{2-x}$O$_4$ and improves the dielectric and attractive properties of ferrite. Samrat Mukherjee et al.,$^{24}$ considered the In$^{3+}$ particle doping on Ni-Co ferrite. The present work considers, Ni$_{0.5}$Co$_{0.5}$Fe$_{2-x}$In$_x$O$_4$ (x=0.0, 0.10, 0.075, 0.050, 0.025) ferrite nanoparticles have been arranged by the sol-gel method. The effect of In$^{3+}$ substitute at the structural and Morphology houses of Nickel-Cobalt ferrite has been examined and pointed out exhaustively.

**EXPERIMENTAL**

Nanocrystalline powder with a synthetic samples Ni$_{0.5}$Co$_{0.5}$Fe$_{2-x}$In$_x$O$_4$ (x=0.0 to 0.025) ferrites were ready using chemical solution deposition method$^{24}$. The Analytical grade reagent was utilized for the blend, for example, Co, Ni, Fe and In nitrate, along with citrus extract (C$_6$H$_8$O$_7$.H$_2$O). The stoichiometric extent of metal nitrate disintegrated in deionized refined water. The molar proportion of 1:3 keeps up with by the expansion of citrus extract arrangement. The pH scale of the subsequent frame-up was modified up to 7, pH by putting liquid alkali, then, at that position the attenuated frame work was heated on the warm plate constantly ageing at 90°C, at last because of auto flaming, brown-hugged detritus was gotten. Pre-arranged examples were sintered at 600°C for 4 hours. The sintering not really settled from TGA/DTA. The power dispersive research of X-beam (EDAX) become carried out to recognize the normal measure of factor and stoichiometry gift in the piece. The X-beam diffraction (XRD) examples of assessments had been recorded at room temperature by means of using Cu–Kα radiation at the Rikagu Miniflag X-beam diffractometer. X-beam diffraction facts had been recorded within the 2θ scope of 20–70° with an inspecting pace of two°/minute. Infrared spectroscopy (IR) estimation changed into finished inside the scope of 800–200 cm$^{-1}$ on a Perkin Elmer infrared Spectrophotometer. The morphological review became finished by using Filtering SEM and became recorded utilizing EDAX Oxford EDAX JEOL–JSM–5600N.

**RESULT AND DISCUSSION**

TGA/DTA

Figure 1 indicates regular (x=0.05) outline of TGA and DSC estimations of arranged powder. TGA and DTA spectra of arranged powder in the whole temperature range 0–1000°C in presence of air. TGA bend indicate mass loss of arranged example because of crumbling of oxides and nitrates. Weight reduction happened in four progressive stages TGA bend it very well may be seen Fig. 1. The top at 103°C was credited by vanishing of assimilated water and the subsequent pinnacle showed up at 144°C ascribed to citrus extract and inorganic mixtures$^{20}$. The sharp abatement in weight reduction top underneath the 330°C is because of metal hydroxides changed over to metal oxide.$^{29}$ The gem immaculateness and the warm soundness at temperature 500 to 550°C. Endothermic top in DTA bend at 450°C might sign the development of solidification at spinel stage.$^{25,30}$ The last calcination temperature is chosen as 600°C for 4 h of the ferrite being scrutinized.

X-ray diffraction (XRD)

The XRD specimen of toughened Ni$_{0.5}$Co$_{0.5}$Fe$_{2-x}$In$_x$O$_4$ (x=0.0.010.0.075,0.050,0.025) are displayed in Fig. 2. XRD pinnacle estimated with Bragg’s shows from (440), (511), (422), (400), (311), and (220) planes relate to the classic construction.
The acquired x-beam design was filed of all the Ni_{0.5}Co_{0.5}Fe_{2-x}In_{x}O_{4} tests which are affirmed the development of a homogenous single stage with cubic spinel structure. (JCPDS NO. 01-088-0380)\textsuperscript{28}. The orchestrated specimen is of FCC construction with space bundle Fd\textsuperscript{3}m. The cubic stage gives information of bigger ionic radii of In(III) particle (0.80 Å) contrasted with the Fe\textsuperscript{3+} particles (0.645 Å)\textsuperscript{31}. The high centralization of indium might twist the gem cross section and creates extra stage on grain limits. This may be because of the supplanting of more modest particles with bigger particles\textsuperscript{20}. The cross-section boundary "a" was determined from the accompanying recipe.

The 'a' was dictated by the connection.\textsuperscript{31}

\[ a = d\sqrt{h^2 + k^2 + l^2} \]  

Where, (h, k, l, and a) are Mill operator lists along with d is interplanar separating and steady cross section (a). Estimated upsides of the lattice steady are summarized up. It is observed that the 'a' continuous up rise from 8.345 Å to 8.378 Å with In\textsuperscript{3+} fixation x. The increments of cross section consistent are arranged in Table 1. The enlargement in cross section consistent are as a result of the variation in ionic radii, the more unpresuming ionic radii of Fe\textsuperscript{3+} (0.645 Å) are supplanted by bigger In\textsuperscript{3+} particle (0.8 Å)\textsuperscript{24}. The X-beam thickness (dx) of the specimen was explained by condition.\textsuperscript{31}

\[ dx = \frac{8M}{Na^3} \]  

Where, M, N, a\textsuperscript{3} are sub-atomic weight of the relating rearrangement, Avogadro's number, volume of unit cell respectively. Upside of 'dx' bring out in Table 1. It is observed that x-beam densities uprise from 5.292 to 5.791 gcm\textsuperscript{-3} with In\textsuperscript{3+} fixation x, this might be on the base that In\textsuperscript{3+} has more noteworthy nuclear mass m (114 amu) supplant maximal unpresuming Fe\textsuperscript{3+} (55.84amu), and its blowoff enlargements in nuclear load with In\textsuperscript{3+} constituents.\textsuperscript{24}

The particle size 't-XRD' of the specimen was estimated utilizing significant pinnacle (311) by Scherrer equation.\textsuperscript{31}

\[ t_{XRD} = \frac{0.9\lambda}{B\cos\theta} \]  

Where, 0, B, \lambda are Bragg point, full width at half greatest, frequency of radiation respectively. Increment of crystal size are arranged in Table 1. Increment of crystal size of the specimen refused with enlarging the In\textsuperscript{3+} particle content , breadth of nanocrystals was decreased for higher In\textsuperscript{3+} particle rate in Ni-Co nano ferrites. Because of the enormous sweep of In\textsuperscript{3+} (0.8 Å) particles concerning change metal particles, indium particles catch the octahedral voids and furthermore forestalled precious stone development during arrangement.\textsuperscript{32}

The weight broadness enlarged from 3.023-3.291 (x=0.0-0.1) along expansion in In\textsuperscript{3+} replacement. The potential justification for expanding densities is the presentation of In\textsuperscript{3+}. The P% of the novel ferrite detect by using the equation.\textsuperscript{31}

\[ P = \left(\frac{dx - dB}{dx}\right) \times 100 \]  

Where, dx is X-ray density and dB is bulk density. Percentage porosity (P\%) decreased 43.863-40.362%, a boom in In\textsuperscript{3+} content material.
The values of the percentage porosity.

The hopping distance at A and B-sites ((L_A) and (L_B)) were calculated by following equation.

\[ L_A = \frac{a}{\sqrt{3}} \]  \hspace{2cm} \text{(5)}

\[ L_B = \frac{a}{\sqrt{4}} \]  \hspace{2cm} \text{(6)}

Hopping lengths (L_A, L_B) the gap between the ions inside the tetrahedral (A) site and octahedral [B] site can be calculated using the relation mentioned someplace else\textsuperscript{22}. The values of expected length are given in Table 1. This shows that the expecting period improved with growing In\textsuperscript{3+} ion awareness. The allied parameters along with tetrahedral and octahedral bond length (d_{Ax} and d_{Bx}), tetrahedral aspect, shared and unshared octahedral edge (d_{AXE}, d_{BXE} and d_{BXEU}) were calculated using experiment value ‘a’, ‘u’ (zero.375 Å) of lattice constant and oxygen positional parameter respectively by substituting in following equation.

\[ d_{Ax} = (u-1/4)a \] \hspace{1.8cm} \text{(7)}

\[ d_{Bx} = [3u^2-(11/4)u+43/64)]^{1/2}a \] \hspace{1.8cm} \text{(8)}

\[ d_{AXE} = (2u-1/2)a \] \hspace{1.8cm} \text{(9)}

\[ d_{BXE} = (1-2u)a \] \hspace{1.8cm} \text{(10)}

\[ d_{BXEU} = [4u^2-3u+(11/16)]^{1/2}a \] \hspace{1.8cm} \text{(11)}

Table 1: Lattice s constant (‘a’), X-ray density(dx), Bulk density(dB), Percentage porosity(P), Particle size (DXRD), Hopping lengths(LA and LB) in Ni\textsubscript{0.5}Co\textsubscript{0.5}Fe\textsubscript{2-x}In\textsubscript{x}O\textsubscript{4} Composition

<table>
<thead>
<tr>
<th>Compound</th>
<th>x</th>
<th>a(obs.) (Å)</th>
<th>Dx(Å)</th>
<th>db (g/cm\textsuperscript{3})</th>
<th>P(%)</th>
<th>DXRD(nm)</th>
<th>LA (Å)</th>
<th>LB(Å)</th>
</tr>
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<tbody>
<tr>
<td>0.0</td>
<td>8.347</td>
<td>5.3853</td>
<td>3.0231</td>
<td>43.8636</td>
<td>13.132</td>
<td>3.622</td>
<td>2.957</td>
<td></td>
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<tr>
<td>0.025</td>
<td>8.359</td>
<td>5.4105</td>
<td>3.1126</td>
<td>42.4709</td>
<td>13.481</td>
<td>3.626</td>
<td>2.960</td>
<td></td>
</tr>
<tr>
<td>0.05</td>
<td>8.365</td>
<td>5.4244</td>
<td>3.1373</td>
<td>42.1633</td>
<td>12.273</td>
<td>3.635</td>
<td>2.968</td>
<td></td>
</tr>
<tr>
<td>0.075</td>
<td>8.369</td>
<td>5.4875</td>
<td>3.1914</td>
<td>41.8422</td>
<td>11.077</td>
<td>3.644</td>
<td>2.975</td>
<td></td>
</tr>
<tr>
<td>0.01</td>
<td>8.378</td>
<td>5.5179</td>
<td>3.2907</td>
<td>40.3627</td>
<td>10.383</td>
<td>3.646</td>
<td>2.977</td>
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</tr>
</tbody>
</table>

The versions of allied parameters with In\textsuperscript{3+} awareness in Ni-Co ferrite is proven in Table 2. The allied parameters are associated with the radii of In\textsuperscript{3+} and Fe\textsuperscript{3+}. The allied parameters are improved with the composition of In\textsuperscript{3+} in Ni-Co ferrite. This boom in aspect lengths arises because of the dopants in doping Incomes and also because of cation distribution inside prepared compositions.

Table 2: x, d_{Ax}, d_{Bx}, d_{AXE}, d_{BXE} and d_{BXEU} of Ni\textsubscript{0.5}Co\textsubscript{0.5}Fe\textsubscript{2-x}In\textsubscript{x}O\textsubscript{4}. (x =0.000,0.100,0.075,0.050,0.025,) Consider, Composition, Tetrahedral bond, Octahedral bond, tetra area, octahedral facet, unshared octahedral area respectively

<table>
<thead>
<tr>
<th>Compound</th>
<th>x</th>
<th>d_{Ax} Å</th>
<th>d_{Bx} Å</th>
<th>d_{AXE} Å</th>
<th>d_{BXE} Å</th>
<th>d_{BXEU} Å</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0</td>
<td>1.8940</td>
<td>2.0379</td>
<td>3.0928</td>
<td>2.8095</td>
<td>2.9528</td>
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<tr>
<td>0.025</td>
<td>1.8967</td>
<td>2.0408</td>
<td>3.0972</td>
<td>2.8135</td>
<td>2.9571</td>
<td></td>
</tr>
<tr>
<td>0.05</td>
<td>1.8981</td>
<td>2.0423</td>
<td>3.0994</td>
<td>2.8155</td>
<td>2.9592</td>
<td></td>
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<tr>
<td>0.075</td>
<td>1.8990</td>
<td>2.0433</td>
<td>3.1009</td>
<td>2.8169</td>
<td>2.9606</td>
<td></td>
</tr>
<tr>
<td>0.1</td>
<td>1.9010</td>
<td>2.0455</td>
<td>3.1042</td>
<td>2.8199</td>
<td>2.9638</td>
<td></td>
</tr>
</tbody>
</table>

Cation distribution

Cation distributions were decided by studying the X-ray diffraction styles' intensity. The determined intensity ratio changed into compared to the calculated depth ratio on this way. Method of Bertaut\textsuperscript{23}. Table 3 suggests the cation distribution of the 600°C samples. The findings display that Ni\textsuperscript{2+}, Co\textsuperscript{2+}, and In\textsuperscript{3+} cations opt to occupy the octahedral positions. The tetrahedral positions are occupied via Fe\textsuperscript{3+} ions[A].\textsuperscript{24} The ionic radius for both tetrahedral and octahedral is (rA and rB) and coordinated sites obeying best cationic distribution had been computed using the following relationships\textsuperscript{22,24}. Both rA and rB are suggested to remain consistent whereas rB increases with a growth.

\[ a_{th} = \frac{8}{3} \sqrt[3]{[\sqrt{r_A} + r_B] + \sqrt{[r_A + r_B]}} \]  \hspace{2cm} \text{(12)}

Where, RO denotes the radius of oxygen (RO=1.32), rB and rA denote the radii of the octahedral [B] and tetrahedral (A) sites, respectively. Table 3 summarizes the values of ‘a\textsuperscript{th}’ and it is able to be proven that the theoretical lattice regular will increase as the quantity of In\textsuperscript{3+} substitution increases\textsuperscript{24}. This follows the equal pattern as the experimentally measured lattice parameter (a). The perfect lattice steady (a\textsuperscript{th}) values had been barely higher than the experimental (a) values, which may
be attributed to the atomic image's center premise that atoms are difficult spheres dependent in a positive sample\(^3\). Using the radius of the oxygen ion \( R_O = 1.32 \) Å, the oxygen positional parameter \( u \) was calculated\(^{31} \).

\[
u = \frac{1}{4} \left( \sum \frac{1}{\sqrt{3}} R_A + \frac{1}{\sqrt{3}} R_B \right)
\]

(13)

Table 3 indicates the values of positional oxygen parameters, and this discovered when Ni-CO ferrite is substituted with \( \text{In}^{3+} \), the value of \( u \) drops. The metallic ions in spinel oxide are lesser than the \( O_2^- \) ions. This result shows that the created spinel lattice differed from the genuine spinel lattice by a small amount\(^{24} \).

### Table 3: \( x, (v_1, v_2), (R_A \text{ and } R_B), (a^0), (u) \) consider as composition, Band role, Cation distribution, Theoretical lattice steady, Oxygen parameter respectively, of \( \text{Cu}_{0.5} \text{Zn}_{0.5} \text{Fe}_{2-x} \text{In}_x \text{O}_4 \) (\( x = 0.000, 0.100, 0.075, 0.050, 0.025 \))

<table>
<thead>
<tr>
<th>Compound ( x )</th>
<th>Cation Distribution</th>
<th>Band Position ( v_1, \text{cm}^{-1} )</th>
<th>( v_2, \text{cm}^{-1} )</th>
<th>Mean ionic radii ( r_A, \text{Å} )</th>
<th>( r_B, \text{Å} )</th>
<th>( a^0, \text{Å} )</th>
<th>( u, \text{Å} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0 (( \text{Fe}^{3+} ))</td>
<td>[( \text{Ni}^{2+} ), ( \text{Co}^{2+}, \text{Fe}^{3+} )]</td>
<td>576.4</td>
<td>354.19</td>
<td>0.670</td>
<td>0.693</td>
<td>8.430</td>
<td>0.3863</td>
</tr>
<tr>
<td>0.025 (( \text{Fe}^{3+} ))</td>
<td>[( \text{Ni}^{2+}, \text{Co}^{2+}, \text{Fe}^{3+}, \text{In}^{3+} )]</td>
<td>580.36</td>
<td>358.6</td>
<td>0.670</td>
<td>0.694</td>
<td>8.435</td>
<td>0.3862</td>
</tr>
<tr>
<td>0.050 (( \text{Fe}^{3+} ))</td>
<td>[( \text{Ni}^{2+}, \text{Co}^{2+}, \text{Fe}^{3+}, \text{In}^{3+} )]</td>
<td>585.64</td>
<td>362.9</td>
<td>0.670</td>
<td>0.696</td>
<td>8.439</td>
<td>0.3861</td>
</tr>
<tr>
<td>0.075 (( \text{Fe}^{3+} ))</td>
<td>[( \text{Ni}^{2+}, \text{Co}^{2+}, \text{Fe}^{3+}, \text{In}^{3+} )]</td>
<td>589.6</td>
<td>395.51</td>
<td>0.670</td>
<td>0.697</td>
<td>8.443</td>
<td>0.3861</td>
</tr>
<tr>
<td>0.1 (( \text{Fe}^{3+} ))</td>
<td>[( \text{Ni}^{2+}, \text{Co}^{2+}, \text{Fe}^{3+}, \text{In}^{3+} )]</td>
<td>583.21</td>
<td>398.64</td>
<td>0.670</td>
<td>0.699</td>
<td>8.448</td>
<td>0.3860</td>
</tr>
</tbody>
</table>

Fourier Transform Infrared Spectroscopy (FT-IR)

FT-IR conveyance spectra of as-gotten ferrite nanoparticles predicted as recurrence scope of 200-800 \( \text{cm}^{-1} \) are displayed in Fig. 3. The FT-IR spectra additionally provide facts approximately the scenario of cations inside the gem\(^{34} \). FT-IR spectra display two full-size agencies at 576-583.21 and 354.19-398.64 \( \text{cm}^{-1} \) that are the trademark band of spinel ferrite. The band 576-583.21 \( \text{cm}^{-1} \) is identified with the extending vibration of the tetrahedral steel-oxygen bond and 354.19-398.64 \( \text{cm}^{-1} \) is relating to the octahedral metal-oxygen bond\(^{24,28,34} \). It has to be noticed that the pressure of the retention top increments whilst the \( \text{In}^{3+} \) particles relocated the spinel structure. The outcome may be clarified via the way that the \( \text{In}^{3+} \) particles may supplant a portion of the \( \text{Fe}^{3+} \) debris in tetrahedral locations and octahedral locales reinforcing the Fe-O extending vibration\(^{20,25,28} \).

Scanning Electron Microscopy, Energy Dispersive X-ray

The morphological investigation of the pre-arranged examples was dictated by utilizing the Checking electron microscopy (SEM) strategy. The run of the mill SEM pictures is displayed in fig. from Fig. 4. The SEM pictures display that the grains have homogenous dispersion and grains are agglomerated. The agglomerate of ferrite powder framed as a result of the warmness effect; this shows the already arranged specimens are the profoundly responsive and uniform dissemination of grains affirming the translucent design of \( \text{In}^{3+} \) doped Ni-Co ferrite\(^{24} \). The pre-arranged samples' agglomeration is additionally because of the attractive communication between the particles\(^{20,24-25} \).
CONCLUSION

\( \text{Ni}_{0.5} \text{Co}_{0.5} \text{Fe}_{2-x} \text{In}_x \text{O}_4 \) nanocrystalline ferrite arranged by Sol-gel strategy. The XRD designs uncovered the development of In-subbed Ni-Co ferrite. The EDAX data obtained from the focal point of In\(^{3+}\) substituted ferrite particle of Ni-Co particles showed the existence of a huge grouping of Ni into the power range of 0.8keV, 7.6keV, and 8.2keV, Co in between 0.4keV, 7.2keV, and 7.6keV and Indium was seen somewhere in the range of 2.8keV, 3.2keV, and 4.2keV, while Iron was found in between the power range of 0.7 to 6.8 keV.

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