Synthesis, Structural, W-H Plot and Size-Strain Analysis of Nano Cobalt Doped MgFe$_2$O$_4$ Ferrite

Rakesh Vishwaroop$^1$, Shridhar N. Mathad$^{2)*}$

$^1$Department of Physics, Jain P.U. College, Davanagere, Karnataka, India
$^2$Department of Physics, K.L.E Institute of Technology-590030, Karnataka, India

Abstract:

In this study we have investigated structural attribute of Co$^{+2}$ doped MgFe$_2$O$_4$. Synthesis of Mg$_{1-x}$Co$_x$Fe$_2$O$_4$ ferrite was carried out using co-precipitation method. The formation of spinal ferrite was confirmed through X-ray diffraction. Lattice parameter found to be 8.376748 Å and crystallite sizes in the range 180-365 Å are observed. Various parameters like dislocation density ($\rho_D$); mechanical properties (strain), Hopping length {tetrahedral site (L$_A$) and octahedral site (L$_B$)}, Bond length (A-O and B-O), and Ionic radii (r$A$ and r$B$) were reported. The W-H plot and Size-Strain plots were extensively studied and the results have been correlated.

Keywords: Synthesis; Structure; Sintering; Spinel.

1. Introduction

Magnetic nano-particles have become subject of intense research because of their applications in high density magnetic recording, in the technological, medical and industrial applications [1]. Spinall ferrite materials attained vast interest because of their unparalleled magnetic, electric and dielectric properties [2-3]. Spinell ferrites with a general chemical formula of MFe$_2$O$_4$, in which M is one or two of divalent metals, such as Co, Mg, Zn, Ni, etc.[3-4].

Magnesium ferrite has cubic structure and it is a soft magnetic n-type semiconductor material. It finds applications in heterogeneous catalysis, adsorption sensors etc. Magnesium ferrite with a chemical formula of MgFe$_2$O$_4$ has an inverse spinel structure, in which half of the trivalent cations occupy the tetrahedral (A) sites and the other half of the trivalent cations and all of the divalent cations fill the octahedral (B) sites [5]. Because of easy fabrications, high efficiencies, thermal stabilities, and low costs of magnesium-ferrites have the broad in scope of applications from low frequencies to microwave frequencies (devices) [6-7]. The excellent magnetic and electrical properties such as high permeability, high electrical resistivity and low dielectric and magnetic losses these ferrites can be used to fabricate as microwave devices like circulators, insulators and phase shifters [8-9].

To accomplish low dielectric losses, reduce the transmission loss [10] cobalt ferrite is used due to high coercivity, high chemical stability and good electrical insulation. Any change in distribution of cations among tetrahedral site and octahedral site by cations substitution have very dominant effects on the physical properties, the substitutions of magnetic or non-magnetic ions alters the spin order which affects the magnetic and electric properties of ferrite structure and greatly affect the ferrite overall properties [11-12]. The

$^{*}$ Corresponding author: physicssiddu@gmail.com
substitution of non-magnetic magnesium ion can modify the properties of cobalt ferrite [12]. The cation distribution according to the earlier reported reveals that magnesium ions exist in both sites (A and B) but have a strong preference for the octahedral (B) site [13]. XRD, FTIR and dielectric studies of Mg–Co nano crystalline ferrites (x=0, 0.05, 0.1, 0.15, 0.2, 0.25) were prepared by the sol–gel method [14].

Numbers of methods to synthesize the ferrites are solid state reactions [15-16], co-precipitation technique [17], microwave processing [18], polymer-assisted route [19], auto combustion [20-21], micro emulsions [22] in reverse micelles [23], sucrose precursor [24].

In this work we report the cobalt doped magnesium ferrite (Mg_{0.85}Co_{0.15}Fe_{2}O_{4}) by simple chemical route by co-precipitation method. Detailed structural properties studied by XRD. The W-H plot and Size Strain Plots (SSP) were extensively studied and the results have been correlated. Dislocation density (ρ_d); mechanical properties (strain), Hopping length {tetrahedral site (L_A) and octahedral site (L_O)}, Bond length (A-O and B-O), and Ionic radii (r_A and r_B) were also reported.

2. Materials and Experimental Procedures

Analytical grade FeCl₃·6H₂O, MgCl₂·6H₂O and CoCl₂·H₂O reagents were weights in molar ratio, in distilled water to produce ionic solution. Ferrite was synthesized from simple low cost co-precipitation method. Ammonia is added drop-wise under constant stirring and a pH of 8 is maintained throughout the reaction. During this method, metal salts converted into hydroxides and subsequent transformation of metal hydroxide into nano Mg_{0.85}Co_{0.13}Fe_{2}O_{4} ferrite.

Precipitate is further powdered using mortar and crusher for one hour. Then the sample is heated to 550 °C for 6 hour in muffle furnace to obtain final nano ferrite powder. The structural characterisation of sample was carried by X-ray diffractometer Bruker AXS D8 Advance diffractometer (Cu–Kα radiation). The schematic diagram of the synthesis method with results observed is shown in Fig. 1.
3. Results and Discussion

3.1 XRD analysis

The XRD pattern of Mg_{0.85}Co_{0.15}Fe_{2}O_{4} was shown in Fig. 2 with peaks (220), (311), (222), (400), (422), (511), and (440) respectively. These plains confirm cubic structure of Mg-Co ferrite. The diffraction maximum from Bragg’s law is prevailed by:

\[ 2d_{hkl} \sin \theta = n\lambda \]  \hspace{1cm} (1)

\[ d = \frac{a}{(h^2 + k^2 + l^2)^{1/2}} \]  \hspace{1cm} (2)

It can be seen that the diffraction peaks are either all even or all odd, which suggests a spinel phase (lattice parameter = 8.376748 Å) for sample and thus validates the cubic structure. The detailed information of sample like lattice parameter (a), and interplanar distances (d) are tabulated in Table I.

| serial no | 2 theta | d value in Å | observed intensity | observed intensity % | standard intensity % | Miller indices | lattice parameter |
|-----------|---------|--------------|--------------------|----------------------|----------------------|----------------|------------------|
| 1         | 33.211  | 2.6954       | 257                | 75.3                 | 10.1                 | 2 2 0          | 8.32564          |
| 2         | 35.617  | 2.51867      | 342                | 100                  | 100                  | 3 1 1          | 8.353483         |
| 3         | 40.934  | 2.20297      | 101                | 29.5                 | 51                   | 4 0 0          | 8.81188          |
| 4         | 43.197  | 2.09265      | 93.3               | 27.3                 | 51                   | 4 0 0          | 8.3706           |
| 5         | 49.384  | 1.84398      | 94.5               | 27.6                 | 9.6                  | 3 3 1          | 8.037722         |
| 6         | 54.109  | 1.69357      | 141                | 41.3                 | 3                    | 4 2 2          | 8.296765         |
| 7         | 57.109  | 1.61151      | 91.8               | 26.8                 | 24.2                 | 5 1 1          | 8.373652         |
| 8         | 62.553  | 1.48371      | 166                | 48.6                 | 41.6                 | 4 4 0          | 8.393131         |

Tab. I Lattice parameter (a), and interplanar distances (d).

Lattice parameter 8.376748

Fig. 2. XRD pattern of nano Mg_{0.85}Co_{0.15}Fe_{2}O_{4} ferrite.
Average Crystallite size (D) is calculated by Debye-Sherrer’s formula [16]:

\[
D = \frac{0.9 \cdot \lambda}{\beta \cdot \cos \theta}
\]  

(3)

\(D\) is size of the particle, \(\lambda\) is the wavelength of x-rays (1.5406 Å), \(\theta\) is Bragg angle for the peak pure diffraction broadening \(\beta\). The calculated average crystallite size (D) of samples is 243 Å.

The distance between magnetic ions (hopping length) in A site (Tetrahedral) and B site (Octahedral) were calculated by using [15-16] the complying relations \{(LA and LB)\}:

\[
L_A = \frac{a \times \sqrt{3}}{4}
\]  

(4)

\[
L_B = \frac{a \times \sqrt{2}}{4}
\]  

(5)

where \(a\) is lattice constant:

\[
A - O = (u - 1/4)a\sqrt{3}
\]  

(6)

\[
B - O = (5/8 - u)a
\]  

(7)

\[
r_a = (u - 1/4)a\sqrt{3} - r(O^{2-})
\]  

(8)

\[
r_b = (5/8 - u)a - r(O^{2-})
\]  

(9)

\[
\text{micro-strain} (\varepsilon) = \frac{\beta \cos \theta}{4}
\]  

(10)

Dislocation Density \((\rho_D) = \frac{1}{D^2}\)

(11)

\[
\rho_D = \frac{15\varepsilon}{aD}
\]  

(12)

The lattice constant \((a=b=c)\), cell volume \((V)\), Dislocation density \((\rho_D)\), micro-strain\((\varepsilon)\), Hopping lengths (tetrahedral site \((L_A)\) and octahedral site \((L_B)\)) bond lengths \((A-O\) and \(B-O)\) and ionic radii \((r_A\) and \(r_B)\) on \(A\)-site and \(B\)-site were systematically order in Table II.

| Lattice parameter (Å) | Volume of unit cell V (e-30) | Hopping length LB (Å) | Hopping length LA (Å) | Bond length A-O (Å) | Bond length A-B (Å) | Ionic Radii rA (Å) | Ionic Radii rB (Å) |
|-----------------------|-----------------------------|-----------------------|-----------------------|---------------------|---------------------|-------------------|-------------------|
| 8.376748              | 587.7956                    | 3.627238              | 2.961628              | 1.813619            | 2.094187            | 0.463619          | 0.744187          |

3.2 Williamson-Hall analysis (W-H plot) and “Size-Strain plot” (SSP) analysis

Assuming the size and strain broadening are additive components of the total integral breadth of a Bragg peak [25]. The distinct angle \((\theta)\) dependencies of both effects laid the basis
for the separation of size and strain broadening in the analysis of Williamson and Hall [15-17].

$$\beta_{hkl} \cos \theta = \frac{K \cdot \lambda}{D} + 4\varepsilon \sin \theta$$  \hspace{1cm} (13)

Fig. 3 shows the variation between the $\beta \cos \theta$ vs. $\sin \theta$ (W-H analysis). The equation (16) represents (linear form) $y = mx + c$ where $m = \text{strain}$ and $c = 1/D$, so that the linear plot of $\beta \cos \theta$ vs. $\sin \theta$ gives the slope as lattice strain ($\varepsilon$) and the intercept as $1/D$.

Fig. 3. Williamson-Hall analysis (W-H plot) for Mg-Co ferrite.

The “size-strain plot” (SSP) is an instrument to interpret the quantity of isotropic nature and micro-strain contribution and the advantage is that less weight is given to data from reflections at high angles, where the precision is usually lower which is shown in Fig. 4. In this approximation, we assume that the “crystallite size” profile is described by a Lorentzian function and the “strain profile” by a Gaussian function [15, 26]. Accordingly, we have:

$$\left( \frac{d_{hkl} \beta_{hkl} \cos \theta}{\lambda} \right)^2 = \frac{3}{4} \frac{\lambda}{D} \left( \frac{d_{hkl} \beta_{hkl} \cos \theta}{\lambda} \right) + \frac{\varepsilon}{2}$$  \hspace{1cm} (14)

Fig. 4. Size-Strain analysis (W-H plot) for Mg-Co ferrite.
In Fig. 4 similarly to the W-H methods, the term \((d_{hkl}^2 \beta_{hkl} \cos \theta)^2\) is plotted with respect to \((d_{hkl}^2 \beta_{hkl} \cos \theta)^2\) for the all orientation peaks of Mg_{1-x}Co_{x}Fe_{2}O_{4} ferrite (x=0.15) ferrite samples with the cubic spinel structure. Crystallite size and lattice strain were also extracted from the XRD data using Williamson-Hall formula through the following equations. In this case, the equivalence between W-H plot and SSP has been reported in Table III. Results of lattice strain and average crystallite size of samples encountered in good agreement with the value obtained from the equations (Table IV).

| Sl. No. | Angle 20 (in degrees) | Angle 0 (in degrees) | cosθ | sinθ | FWHM β (in radians) | βcosθ | D (Å) | Dislocation Density \(\rho D\) X 10 \(^{-4}\) | Micro Strain \(\epsilon\) | d | \(d^*(d\beta\cos\theta)\) |
|--------|-----------------------|----------------------|------|------|---------------------|--------|-------|-----------------------------|----------------|----|---------------------|
| 1      | 33.211                | 16.6055              | 0.958263 | 0.285877 | 0.005936 | 0.005689 | 243.7386 | 1.68326 | 0.001422 | 2.6954 | 0.000235 | 0.041329 |
| 2      | 35.617                | 17.8085              | 0.952047 | 0.309951 | 0.006548 | 0.006234 | 222.4325 | 2.02117 | 0.001558 | 2.51867 | 0.000246 | 0.039544 |
| 3      | 49.384                | 24.692               | 0.908497 | 0.417891 | 0.007333 | 0.006662 | 208.1207 | 2.30871 | 0.001666 | 1.84398 | 0.000151 | 0.022653 |
| 4      | 54.109                | 27.0545              | 0.890492 | 0.454999 | 0.005814 | 0.005177 | 267.8021 | 1.39435 | 0.001294 | 1.69357 | 7.69E-05  | 0.01485  |
| 5      | 62.555                | 31.2765              | 0.854563 | 0.519348 | 0.006615 | 0.00567  | 244.546  | 1.67216 | 0.001417 | 1.48371 | 7.69E-05  | 0.012482 |

Results of lattice strain and average crystallite size of samples encountered in good agreement with the value obtained from the equations (Table IV).

| Tab. IV Calculated values of crystallite size, micro strain and dislocation density using W-H plots, SSP and standard formula. |
|--------------------------------------------------------------------------------------------------------------------------------|
| **Crystallite size \(A^3\)** | **Micro strain** | **Dislocation density \(\rho\)** |
| From W-H graph | From SSP | From formula \(\epsilon = \frac{d^*(d\beta\cos\theta)}{\rho D}\) | From formula \(\epsilon = \frac{d^*(d\beta\cos\theta)}{\rho D}\) | \(\rho = \frac{1}{\rho D}\) | \(\rho = 15\varepsilon /\mu D\) |
| 364 | 184 | 237 | 0.001325 | 0.0493 | 0.001473 | 1.81X10\(^{-15}\) | 1.111 X 10\(^{-15}\) |

### 3.3 Texture analysis

The reflection intensities from each XRD pattern contain information related to the preferential or random growth of polycrystalline material, which is studied by calculating texture coefficient \(TC_{(hkl)}\) for all planes using [15-17]:

\[
TC_{(hkl)} = \frac{I_{(hkl)}}{I_{o(hkl)}} \left( \frac{1}{\sum N_{(hkl)}} \right) \quad (15)
\]

| Tab. V Texture analysis of Sample. |
|-----------------------------------|
| **Sl. No.** | **Miller indice (Planes)** | **Texture analysis (TC)** |
|-------|----------------|------------------|
| h | k | l |
| 1 | 2 | 0 | 3.432294 |
| 2 | 3 | 1 | 0.495231 |
| 3 | 4 | 0 | 0.281602 |
| 4 | 1 | 1 | 0.262181 |
| 5 | 3 | 1 | 1.392837 |
| 6 | 5 | 1 | 0.55253 |
| 7 | 4 | 0 | 0.583325 |

where \(I_{(hkl)}\) is the measured intensity of X-ray reflection, \(I_{o(hkl)}\) is the corresponding standard intensity and \(N\) is the number of reflections observed in the XRD pattern. Texture coefficient is higher than one indicates preferential orientation and also indicates the abundance of grains...
along the given (hkl) plane. TC(220) has relatively higher value 3.43 than other planes indicating higher orientations of crystallites along these particular planes. TC for different (hkl) planes is demonstrated in Table V. It is observed that, preferential orientation (abundance of grains) in (220) plane direction.

The stacking fault probability was calculated by measuring the peak shift and tangent values of diffracting angle:

$$\alpha = \left[ \frac{2\pi^2}{45\sqrt{3}} \right] \frac{\Delta 2\theta}{\tan \theta_{hl}}$$

(16)

$\alpha$- stacking fault coefficient, $\Delta 2\theta$- difference in standard and observe $2\theta$ values. The detailed analysis of stacking fault probability is shown in Table VI and observed to be for this ferrite as 0.0735.

The growth mechanism of ferrite sample can be estimated by calculating the standard deviation using the equation [17]:

$$\sigma = \sqrt{\frac{\sum I_{hl}^2 - (\sum I_{hl}^2/2)}{N}}$$

(17)

where $I_{hl}$ stands for relative intensity of the (hkl) plane. The estimated standard deviation, $\sigma$, in the relative intensity values was calculated and tabulated in Table VII from the values of the five strongest lines, excluding the line with $I_{hl} = 100$. The calculated value of $\sigma$ is 45.25, which appears to be relatively depleted showing that heterogeneous nucleation, desorption and adsorption are recessive and the homogeneous nucleation looks predominant [28].

| Observed Angle 20 | Observed Angle 20 in rad | Calculated d (Å) | Calculated Angle 0 | Calculated Angle 20 | $\Delta 2\theta$ | $\tan \theta$ | h | k | l | Stacking fault coefficient ($\alpha$) |
|------------------|-------------------------|------------------|-------------------|-------------------|----------------|-------------|---|---|---|-------------------------------|
| 33.211           | 0.579532                | 2.6934           | 0.263658          | 0.527317          | 0.052115       | 2           | 2 | 2 | 0 | 0.044359                      |
| 35.617           | 0.621517                | 2.51867          | 0.310567          | 0.621133          | 0.621133       | 3           | 1 | 1 | 1 | 0.489884                      |
| 40.934           | 0.714298                | 2.20297          | 0.377464          | 0.754929          | 0.040631       | 0.373152     | 4 | 0 | 0 | 0.027581                      |
| 43.197           | 0.753788                | 2.09265          | 0.377464          | 0.754929          | 0.001141       | 0.395815     | 4 | 0 | 0 | 0.00073                       |
| 49.384           | 0.861751                | 1.84398          | 0.413299          | 0.826899          | 0.035152       | 0.459681     | 3 | 3 | 1 | 0.01937                       |
| 54.109           | 0.942202                | 1.69357          | 0.468331          | 0.936661          | 0.007541       | 0.510612     | 4 | 2 | 2 | 0.003741                      |
| 57.109           | 0.996552                | 1.61151          | 0.499292          | 0.998584          | 0.002032       | 0.544066     | 5 | 1 | 1 | 0.000946                      |
| 62.533           | 1.09155                 | 1.48371          | 0.548292          | 1.096584          | 0.005034       | 0.607307     | 4 | 4 | 0 | 0.002099                      |

Average = 0.073589

| Observed Intensity % | I* | I*I | I*I/2 | Standard Deviation |
|----------------------|----|-----|-------|-------------------|
| 24                   | 576 | 288 | 46.38819 |
| 26.8                 | 718.24 | 359.12 | 46.23462 |
| 27.3                 | 745.29 | 372.645 | 46.20536 |
| 27.6                 | 761.76 | 380.88 | 46.18753 |
| 29.5                 | 870.25 | 435.125 | 46.06994 |
| 41.3                 | 1705.69 | 852.845 | 45.15412 |
| 75.3                 | 5670.09 | 2835.045 | 40.52721 |

Standard Deviation = 45.25242
4. Conclusion

The nano cobalt substituted Mg-ferrite (Mg$_{0.85}$Co$_{0.15}$Fe$_2$O$_4$) was successfully synthesized by a coprecipitation method. Structural properties were investigated by XRD analysis shows cubic single phase spinel with lattice parameter 8.376748 Å and crystallite-size (D) is 243 Å. We have also discussed dislocation density ($\rho_D$), mechanical properties (strain), hopping length {tetrahedral site (LA) and octahedral site (LB)}, bond length (A-O and B-O), ionic radii (rA and rB) and stacking fault probability (α) of Mg$_{0.85}$Co$_{0.15}$Fe$_2$O$_4$ sample. The W-H plot and size strain plots were extensively studied and the results have been correlated. Thus Low-cost chemical method (co-precipitation) technique is a favourable way to obtaining homogeneous nano Mg$_{1-x}$Co$_x$Fe$_2$O$_4$ ferrite.

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Саметак: У овом раду смо испитивали структуру MgFe_{2}O_{4} допираног са Co^{2+}. Синтеза Mg_{1-x}Co_{x}Fe_{2}O_{4} ферита је изведена ко-предципитацијом. Формирање спинела је потврђено рендгенском дифракцијом. Параметри решетке су 8.376748 Å и величина кристалита је у опсегу 1\text{80}-365 Å. Такође су дате вредности за различите параметре као што су густина дислокација (\rho_D); механичка својства (напрезање), Хопингова дужина \{тетраедарски положај (L_A) и октаедарски положај (L_B)\}, дужина везе (A-O и
B-O), и јонски радијуси (rA and rB). Криве W-H и величина-напрезање су детаљно испитане и резултати корелисани.

Кључне речи: синтеза, структура, синтеровање, спинел.

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