



## Structural and magnetic properties of nanocrystalline stannic substituted cobalt ferrite

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### ABSTRACT

The structural and magnetic properties of the spinel ferrite system  $\text{Co}_{1+x}\text{Fe}_{2-2x}\text{Sn}_x\text{O}_4$  ( $x=0.0-1.0$ ) have been studied. Samples in the series were prepared by the ceramic technique. The structural and microstructural evolutions of the nanophase have been studied using X-ray powder diffraction and the Rietveld method. The refinement result showed that the type of the cationic distribution over the tetrahedral and octahedral sites in the nanocrystalline lattice is partially an inverse spinel. Far infrared absorption spectra show two significant absorption bands, around  $600\text{ cm}^{-1}$  and  $425\text{ cm}^{-1}$ , which are respectively attributed to tetrahedral (A) and octahedral [B] vibrations of the spinel. Scanning Electron Microscopy (SEM) was used to study surface morphology. SEM images reveal particles in the nanosize range. The transmission electronic microscope (TEM) reveals that the grains are spherical in shape. TEM analysis confirmed the X-ray results. The magnetic properties of the prepared samples were characterized by using a vibrating sample magnetometer.

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### 1. Introduction

Nanocrystalline materials are the focus of recent research due to their potential applications and interesting physics involved in them. The ferrite materials substituted with different cations prepared by different techniques have become important from both the fundamental and application point of view [1,2]. The properties of ferrite are very much sensitive to methodology adopted for their synthesis, preparative parameters, initial ingredients, etc. Any change in cation distribution also results into an unexpected electrical and magnetic behavior.

Cobalt ferrite,  $\text{CoFe}_2\text{O}_4$ , is a hard magnetic material [3], which finds a number of applications in heterogeneous catalysis, adsorption, sensors, and in magnetic technologies. Solid-state processing techniques are very suitable for the preparation of nanocrystalline ferrite powders exhibiting many of the useful properties listed above [4–7]. Ferrites have the general formula  $(\text{M}_{1-a}\text{Fe}_a)[\text{M}_a\text{Fe}_{2-a}]\text{O}_4$ , where  $a$  represents the degree of inversion (defined as the fraction of (A) sites occupied by  $\text{Fe}^{3+}$  cations). The divalent metal element M (Mg, Zn, Mn, Fe, Co, Ni or mixture of them) can occupy either tetrahedral (A) or octahedral [B] sites in the cubic, spinel-type structure. The magnetic properties of a spinel ferrite

are strongly dependent on the distribution of the different cations among (A) and [B] sites.

Physical properties of materials depend upon their microstructure and, therefore, its knowledge is an important prerequisite to controlling material's performance. Rietveld analysis [8–10] has been adopted in the present study to determine the microstructural parameters of nanocrystalline  $\text{CoFe}_2\text{O}_4$ . The analysis aims to (i) characterizing the prepared materials in terms of microstructural parameters such as crystallite size and lattice strain and (ii) estimating the distribution of cations among (A) and [B] sites in the spinel lattice.

### 2. Experimental

#### 2.1. Samples preparation

The nanocrystalline  $\text{Co}_{1+x}\text{Fe}_{2-2x}\text{Sn}_x\text{O}_4$  ( $x=0.0-1.0$ ) was prepared by the ceramic technique. Accurately weighed powders of  $\text{CoO}$  (99.9% purity),  $\text{Fe}_2\text{O}_3$  (99.9% purity) and  $\text{SnO}_2$  (99.9% purity) were mixed together using triple distilled water as mixing medium in mortar, then dried at  $200\text{ }^\circ\text{C}$  for 2 h, and then milled for 4 min in a Blender (18000 rpm). The milled powder were pre-sintered to  $500\text{ }^\circ\text{C}$  for 3 h, then rapidly cooled to room temperature, and finally re-milled again for 4 min. The pre-firing mixture was sintered at  $1000\text{ }^\circ\text{C}$  for 3 h, after that re-milled again for 2 min. All sintering processes are carried in open air.

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## 2.2. Characterization techniques

The X-ray powder diffraction patterns of the samples were collected on a Bruker Axs-D8 Advance powder diffractometer with a goniometer using Cu K-Alpha radiation. The diffracted intensities were collected in step-scan mode (step size  $2\theta=0.02^\circ$ ; counting time 2 s) in the angular range  $2\theta=10^\circ$ – $80^\circ$ . To correct instrumental broadening a Si standard [11] was used. Rietveld analysis [12] was performed using the program Fullprof; it is designed to refine simultaneously both the structural (lattice cell constants and atomic positions and occupancies) and microstructural parameters (crystallite size and lattice strain). The Fourier Transform Infrared Spectroscopy (FTIR) spectra of the samples were recorded as (KBr) disks in the range  $400$ – $4000\text{ cm}^{-1}$  using a spectrophotometer (Jasco FT/IR-6100). Microstructure characterization of the powder has also been done using electron microscopy (SEM, JEOL JSM 5410, Japan, and TEM, JEOL JEM 1230, Japan). A vibrating sample magnetometer (9600-1 VSM L.D.J, USA) is used for the magnetic measurement.

## 3. Results and discussion

### 3.1. XRD analysis

The X-ray diffraction patterns of polycrystalline  $\text{Co}_{1+x}\text{Fe}_{2-2x}\text{Sn}_x\text{O}_4$  ( $x=0.0$ – $1.0$ ) prepared by the ceramic technique at  $1000^\circ\text{C}$  for 3 h are shown in Fig. 1. The X-ray diffraction analysis by computer search matching for nanocrystalline  $\text{Co}_{1+x}\text{Fe}_{2-2x}\text{Sn}_x\text{O}_4$  showed that all samples consisted of single phase and they do not contain any reflection from starting phases. Graphical representation of the variation of the  $(h^2+k^2+l^2)^{1/2}$  vs.  $(1/d)$  for  $\text{Co}_{1.4}\text{Sn}_{0.4}\text{Fe}_{1.2}\text{O}_4$  and  $\text{Co}_{1.6}\text{Sn}_{0.6}\text{Fe}_{0.8}\text{O}_4$  are shown in Fig. 2.

### 3.2. Refinement of XRD data

FULLPROF Rietveld software program has been used for Rietveld XRD data analysis for  $\text{CoFe}_2\text{O}_4$ . All refinements were performed in space group: Fd-3m. Atomic scattering factor for fully ionized atoms  $\text{Co}^{2+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Sn}^{4+}$  and  $\text{O}^{2-}$  were taken from the international table for crystallography volume C (1992).

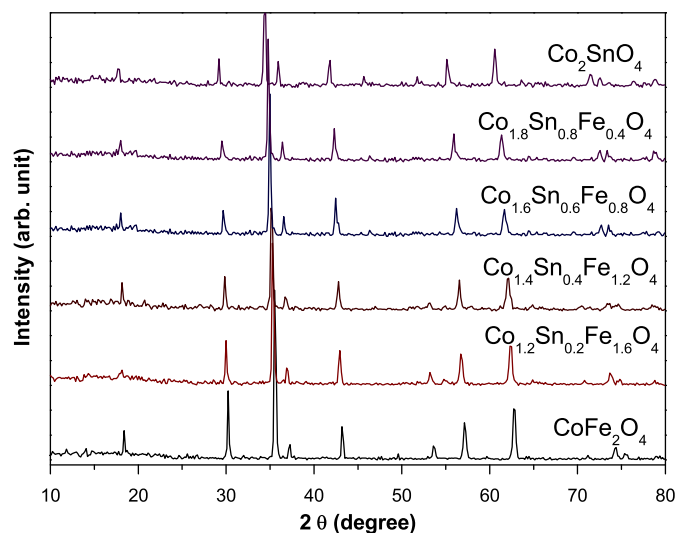


Fig. 1. X-ray diffraction pattern for  $\text{Co}_{1+x}\text{Fe}_{2-2x}\text{Sn}_x\text{O}_4$  prepared by the solid state method sintering at  $1000^\circ\text{C}$ .

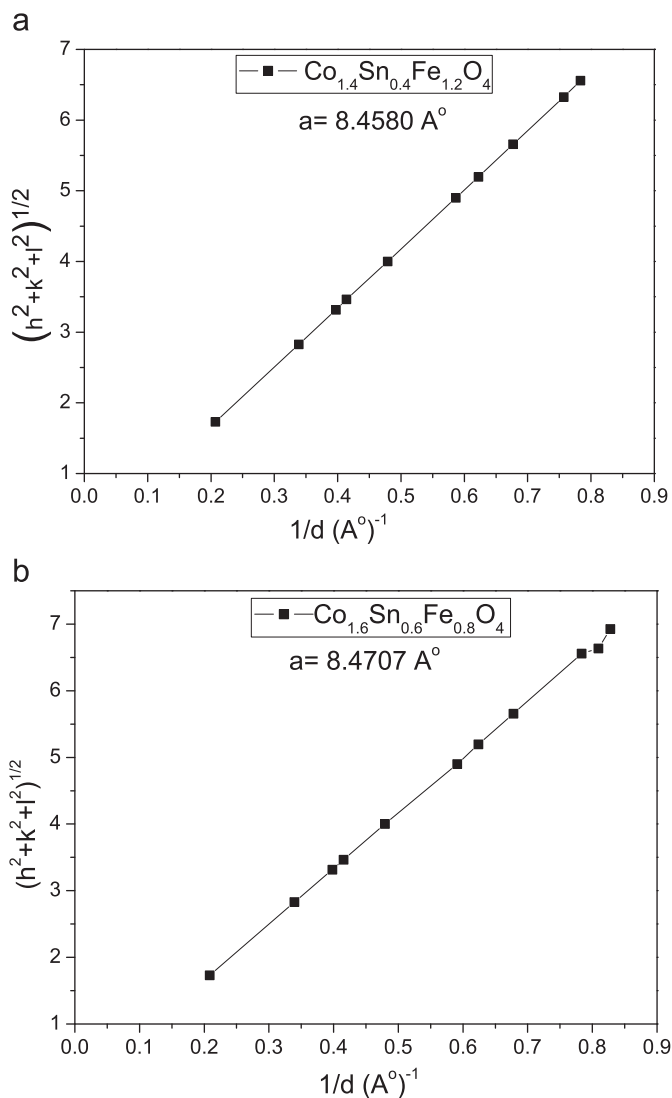


Fig. 2.  $(h^2+k^2+l^2)^{1/2}$  vs.  $(1/d)$  for (a)  $\text{Co}_{1.4}\text{Fe}_{1.2}\text{Sn}_{0.4}\text{O}_4$  and (b)  $\text{Co}_{1.6}\text{Fe}_{0.8}\text{Sn}_{0.6}\text{O}_4$  prepared by the ceramic technique.

Starting cell parameter was taken from the results of the precedent section and the oxygen parameter starting values were taken as the ideal atomic position of the ion  $\text{O}^{2-}=0.25$ . In each refinement, a total of more than 20 parameters were refined: zero shift, scale factor, back ground coefficients, three lattice constants, asymmetry parameter, oxygen parameter ( $U$ ), temperature factor, and parameters for the full width at half maximum.

The Rietveld plots of the refinements for  $\text{Co}_{1.4}\text{Sn}_{0.4}\text{Fe}_{1.2}\text{O}_4$  and  $\text{Co}_{1.6}\text{Sn}_{0.6}\text{Fe}_{0.8}\text{O}_4$  are shown in Fig. 3. In the figure the observed intensity data are plotted in the upper field as points. The calculated patterns are shown in the same field as a solid-line curve. The difference, observed minus calculated, is shown in the lower field. The short vertical bars in the middle field indicate the positions of possible Bragg reflections. Table 1 depicts the refinement, fitting parameters, lattice parameter  $a$  (Å) and oxygen parameter for the prepared samples.

The lattice parameter as shown in Fig. 4 increases as the concentration of Sn cation increases. This means that the lattice parameters of these spinel compounds are closely related to the size of the cations. The reason is that, the atomic radius of  $\text{Sn}^{4+}$  (0.745 Å) is larger than  $\text{Fe}^{3+}$  (0.645 Å) [13]. Moreover, the  $\text{Sn}^{4+}$  has a strong site preference for B-site [14]. The partial replacement of  $\text{Fe}^{3+}$  by  $\text{Sn}^{4+}$  will cause the stretching of unit cell.

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