

X-Ray Diffraction Studies of Mn –Si Spinel Ferrite

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ABSTRACT

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The structural properties of Mn-Si spinel ferrites reported in this present paper. Mn-Si spinel Ferrite with composition $Mn_{1+x}Si_xFe_{1-2x}O_4$ ($x=0.0$ to 0.3 in the step of 0.1) were prepared by standard Sol –Gel method using A.R grade $MnFe_2O_4$. The prepared samples were characterized by using X-ray (XRD) X-ray density, Lattice constant, and hopping lengths (LA and LB), allied parameters such as tetrahedral and octahedral bond length (d_A and d_B), tetrahedral edge, shared and unshared octahedral edge were calculated using X-ray diffraction data. Hopping lengths and allied parameters varied with Mn-Si. The synthesized powder samples were annealed at 600°C for 4h. X-ray diffraction data were used to evaluate the structure of the prepared samples.

Keywords - XRD, sol-gel method, Ferrite, hopping length.

I. INTRODUCTION

Rationale of the study:

Ferrites are widely used magnetic materials due to their high electrical resistivity, low eddy currents and dielectric loss. These materials are extensively used in microwave devices, computer memory chips, magnetic recording media etc. Technically, fine ferrites have been of interest due to their applications for preparation of high density ferrites as suspension materials in ferrofluids at low temperature. High frequency applications of these materials find use in the fabrication of radiofrequency coils, transformer cores, and rod antennas. Ferrites should also be mechanically strong to resist damage during machining and assembly of parts. Manganese-zinc ferrites belong to the group of soft ferrite materials

characterized by high magnetic permeabilities and low losses (1) Spinel ferrites have been the subject of great interest for the past five decades, because of their wide range of applications in transformers, inductors, choke coils, noise filters magnetic recording heads, and so forth [2]. Manganese ferrite is early known to be a mixed inverse spinel, and the degree of inversion mainly depends upon the method of preparation. The presence of nonmagnetic ions in these spinel ferrites is found to alter their magnetic and electronic properties. The addition of metal cations such as trivalent or tetravalent influences the electronic and magnetic properties of the ferrite system [3–6]. Soft magnetic ferrites with spinel structure are materials with many applications in electronic and telecommunication industries. The properties of these materials are highly sensitive to

the cation distribution, which in turn depends on the preparation and sintering conditions (7). Recent interest in the study of several spinel-type ferrites has focused on the development of nanosized particles at low temperatures by different chemical synthesis techniques, in view of the potential application of these nanosized magnetic materials in different technological areas, as well as studies of the structural, electrical and magnetic properties of nanoferrite materials.

The nanostructured magnetic particles have different properties from the corresponding bulk material due to their reduced size and effect of magnetic interaction between particles. Magnetic nanoparticles are gaining importance due to their potential applications in high-density magnetic recording, magnetic fluid, biomedical and microwave applications etc. During the last few decades, ferrites have been studied extensively because of their importance in basic as well as applied research. Ferrite, i.e. ferromagnetic cubic spinel, possesses the combined properties of magnetic materials and insulators. The spinel ferrites (MFe_2O_4) belong to an important class of magnetic material because of their remarkable magnetic and electrical properties particularly in the radio frequency region, physical flexibility, high electrical resistivity, mechanical hardness, and chemical stability (8).

II. OBJECTIVE OF STUDY

- 1) To know synthesis spinel ferrite systems by sol-gel auto combustion technique in this present work two compositions were prepared by using sol gel auto combustion technique. The final powders are sintered at four various temperatures.
- 2) To analyse the crystal structure of all the samples of both the series were confirmed by using XRD data structural parameter lattice constant X-ray density particle size, bulk density

- 3) Transmission Electron Microscopy particle size of all the samples is calculated from XRD data and the calculated particle size.
- 4) Magnetic properties Room temperature magnetic properties were studied by using vibrating sample magnetometer.
- 5) Electrical properties dc electrical resistivity & dielectric properties like dielectric constant, dielectric loss & dielectric loss tangent were studied as function of temperature and frequency by using two probe method.

III. HYPOTHESIS

Ferrites and ferromagnetic material are magnetic oxides its Important for structural, electrical and magnetic properties variety of application. This property strongly depends upon the method of preparation. The effect of varying synthesis condition such as chelating fuel to nitrate ratio, effect of PH, sintering time/temperature etc. ferrite in the form of nanocrystalline powder, nanowires, nanotubes and thin film of spinel hexaferrite, and bismuth ferrite, possible mechanisms that affect the structure formation and properties of ferrite along with their application in various technological.

Thin Film Bulk materials are widely used in electroacoustic devices, motors, electricity generators, etc. On the other hand, thin films of ferrites are widely applicable in the fabrication of magnetic and magneto-optic devices, such as high-density recording media because of their excellent chemical stability, low corrosion, moderate mechanical durability, and considerably high uniaxial magnetic anisotropy. In the thin film form, ferrites were proposed to be the important materials for nonreciprocal microwave devices because of its high electrical resistivity and frequency dependent permittivity. Surface morphology of the ferrite thin films is crucial to the signal-to-noise ratio (SNR) in high density magnetic recording media. Precise control over morphologies of bulk/thin film materials

is an important aspect from the point of view of their applications and also important in material science research. It is a result of atom immigration in nonequilibrium or near equilibrium state. (9).

IV. METHODOLOGY

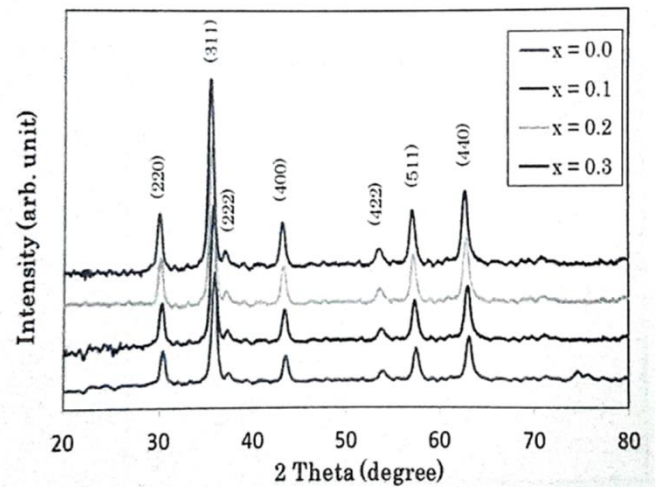
Nanoparticles of the composition $Mn_{1+x}Si_xCrFe_{1-2x}O_4$ were prepared by using sol-gel auto-combustion method. Manganese nitrate, silicon nitrate, chromium nitrate and citric acid ($C_6H_8O_7.H_2O$) were used as starting materials. An aqueous solution of stoichiometric amount of Mn^{2+} nitrate, Si^{4+} nitrate, Cr^{3+} nitrate, Fe^{3+} nitrate was reached with citric acid in 1:3 molar ratio. The pH of the solution was increased to 7 by the addition of ammonia solution to complete the reaction of the Mn-Si-Cr Ferrite. The solution was evaporated very slowly over a period of four hours to dryness. Viscosity and color changed as the sol, turned into puffy, porous dry gel. As soon as the solvent removal was complete the dried, precursor went under a self-ignition reaction to form a very fine powder known as synthesized powder. The synthesized powder thus obtained was treated in furnace at $400^\circ C$ for 6 to remove the residual.

STRUCTURAL ANALYSIS

The X-ray diffraction (XRD) patterns of the $Mn_{1+x}Si_xCrFe_{1-2x}O_4$, ferrite system with $X= 0.0-0.3$, in the steps of 0.1 are shown in fig1. The XRD pattern for $Mn_{1+x}Si_xCrFe_{1-2x}O_4$, indicate well-defined peaks of crystalline FCC phase which confirm spinel cubic structure formation for the samples. No additional impurity reflections were observed ensuring the phase purity. The inter-planer spacing 'd' values calculated from the XRD data is presented in table 1.1. It is observed from XRD patterns that the diffraction peak shifts towards to lower angle (2θ) with Mn-Si substitution. This shift in the diffraction angles shows that the lattice constant of $MnCrFeO_4$ is disturbed with Mn - Si substitution. the lattice constant (a) of

all the samples was determined by using the following equation.

Fig.1: XRD patterns of $Mn_{1+x}Si_xCrFe_{1-x}O_4$ (x=0.0, 0.1, 0.2 and 0.3)



The distance between magnetic ions (jump lengths) in tetrahedral A- site and octahedral B-site i.e. 'La' and 'L_β' respectively is given by :

$$La = a\sqrt{\frac{3}{4}} \quad (1.1)$$

$$L\beta = a\sqrt{\frac{2}{4}} \quad (1.2)$$

The variation of hopping length with Mn-Si substitution is shown in Fig.2 The value of hopping length is presented in Table 1.2 It can be seen from Fig.3 and table 1.3 that the distance between the magnetic ions (jump length) increased with Mn-Si substitution. This behavior of jump length with x is analogous with the behavior of lattice constant with Mn-Si substitution. This variation may be attributed to the difference in the ionic radii of the constituent ions, which makes the magnetic ions become larger to each other and the hopping length increase

Using the experimental values of lattice constant 'a', oxygen positional parameter 'u' and substituting using the following equations, the allied parameters such as tetrahedral and octahedral bond length (d_{ax} and $d_{\beta x}$), tetrahedral edge, shared and unshared octahedral edge (d_{axe} and $d_{\beta x}$ and $d_{\beta xe}$) were calculated :

$$d_{ax} = a\sqrt{3}(u - 1/4) \quad (1.3)$$

$$d_{\beta x} = a[3u^2 - (11/4)u + 43/64]^{1/2} \quad (1.4)$$

$$d_{ax} = \sqrt{2}(2u - 1/2) \quad (1.5)$$

$$d_{\beta xe} = a\sqrt{2}(1 - 2u) \quad (1.6)$$

shows the variation in all the allied parameter. It is observed that allied parameters increased with increase in Mn-Si substitution. This could be related to the larger average ionic radius of Mn-Si ions compared to Fe³⁺ ions.

Table 1.1

Hopping lengths (L_A and L_B) of $Mn_{1+x}Si_xCrFe_{1-x}O_4$ ($x=0.0, 0.1, 0.2$ and 0.3)

Comp. x	Hopping lengths	
	L_A (Å)	L_B (Å)
0.0	3.5708	2.9155
0.1	3.5829	2.9254
0.2	3.6019	2.9409
0.3	3.6185	2.9545

Table 1.2

Tetrahedral bond (d_{AX}), octahedral bond (d_{BX}), tetra edge (d_{AXE}) and octahedral edge (d_{BXE}) (shared and unshared) of $Mn_{1+x}Si_xCrFe_{1-x}O_4$ ($x=0.0, 0.1, 0.2$ and 0.3)

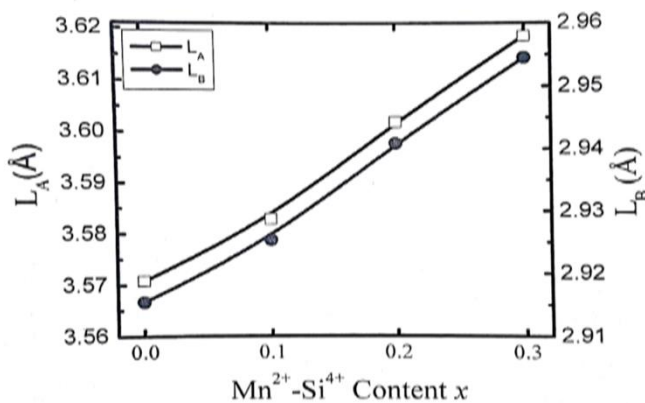


fig 1.3

Comp. 'x'	d_{AX} (Å)	d_{BX} (Å)	Tetra edge (Å)		Octa edge d_{BXE} (Å)	
			d_{AXE}	Shared	Unshared	
0.0	1.871	2.015	3.055	2.775	2.923	
0.1	1.877	2.022	3.065	2.785	2.933	
0.2	1.887	2.033	3.082	2.799	2.948	
0.3	1.896	2.042	3.096	2.812	2.962	

In this work we have choose divalent ferromagnetic Mn²⁺ ion as Me²⁺ and tetravalent magnetic Si⁴⁺ ion as

Me⁴⁺. The addition of tetravalent ions like Ti⁴⁺, Ge⁴⁺, Si⁴⁺, influences the structural, magnetic and transition properties of the system [10-13].

V. CONCLUSIONS

Mn-Si substituted MnCrFeO₄ nano-crystalline ferrite with a chemical formula Mn_{1+x}Si_xCrFe_{1-2x}O₄ (x = 0.0 to 0.3, in the step of 0.1) were prepared by using sol-gel –auto –combustion technique. The analysis of X-ray diffraction patterns reveals the formation of single-phase cubic spinel structure. The lattice constant of MnCrFeO₄ increased with Mn –Si substitution due to the difference between ionic radii of the ions. The X-ray density decreased with Mn -Si substitution due to change in molecular weight of the samples. The particle size estimated showed an increasing trend with the Mn- Si substitution because the replacement of Fe³⁺ by Mn²⁺-Si⁴⁺ ions weaken the sublattice interaction.

Recommendations:

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