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ORIGINAL ARTICLE

STRUCTURAL PROPERTIES OF $MgFe_{2-x}Cr_xO_4$ SYNTHESIZED BY WET-CHEMICAL CO-PRECIPITATION METHOD

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Abstract:

The structural properties of Cr substituted Magnesium ferrites having general formula $MgFe_{2-x}Cr_xO_4$ (where x = 0.0, 0.2, 0.4, 0.6, 0.8 and 1.0) were synthesized by the wet chemical co-precipitation method were investigated. The samples were annealed at 8000C for 12 h and were studied by means of X-ray diffraction, Scanning electron microscope (SEM), particle size, cation were also studied. The X-ray analysis showed that all the samples had single-phase cubic spinel structure. The nano size nature of the particles was confirmed from the values of particle size obtained from XRD and SEM data. The variation of lattice constant with Zn concentration deviates from Vegard's law XRD analysis proves the formation of single phase cubic spinel structure. Cation distribution data shows that Cr3+ occupies octahedral B site where as Mg^{2+} and Fe^{3+} occupies tetrahedral A site.

KEYWORDS:-

Co-precipitation method, Ferrite, SEM, structural properties, XRD.

INTRODUCTION

Polycrystalline soft ferrite materials are used for microwave applications, high quality filter, antenna rods, transformer core, sensors etc. owing to their high electrical resistivity, low eddy current losses and chemical stability [1, 2]. Spinel ferrites can be described by the formula MFe_2O_4, where M is a divalent metal ion like Co, Ni, Zn, Cd, etc. The magnetic nano-particles of spinel ferrite are of great technical importance because of their use in magnetic field information storage, medical diagnostic etc. [3, 4]. It is interesting that the desired electrical and magnetic properties of spinel ferrites can be tailored by controlling the different types and amount of substituent and also the method of preparation. Various preparation techniques have been used for the synthesis of fine particles of spinel ferrites, which exhibits novel properties that are differed from the properties in bulk. Non-conventional methods such as micro-emulsion, thermal decomposition, sol-gel, hydrothermal etc methods have been widely used. Ultrafine particles of ferrite can also be prepared by simple chemical co-precipitation method. Among the spinel ferrites magnesium ferrite possesses good electrical and magnetic properties and is of great interest from the point of view of their applications in many fields. Magnesium ferrite in bulk crystal has an inverse spinel structure; where the tetrahedral (A) sub-lattice contains half of the Fe^{3+} ions and the B sub-lattice contain the other half together with all of the Mg^{2+} ions. Further, it is interesting that $MgFe_2O_4$ show magnetism even though Mg^{2+} ions are non-magnetic. This may be due to the inverse spinel structure of magnesium ferrite. The substitutional study of Cr^{3+} ions in magnesium ferrite prepared by chemical route is less studied [5]. Cr^{3+} ions have strong preference for octahedral [B] site whereas Mg^{2+} ions have preference for both tetrahedral and octahedral site. Further the effect of nanosize particles on the properties of Mg-Cr spinel ferrite has not been investigated in detail. Therefore, it will be interesting to study the structural, electrical

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and magnetic properties of chromium substituted magnesium ferrite prepared by wet chemical co-precipitation method.

In this paper our results on the structural magnetic properties of $MgFe_{2-x}CrO_4$ (x=0.0,0.2,0.4,0.6,0.8,1.0.) prepared by wet chemical co-precipitation method.

EXPERIMENTAL DETAIL

The samples of $MgFe_{2-x}CrO_4$ with varying x (x = 0.0 to 1.0) were synthesized by wet-chemical co-precipitation technique. AR grade sulphates of the constituent ions(Mg^{2+},Cr^{3+},Fe^{3+}) were used for the preparation of Mg-Cr spinel ferrite system. The solutions of sulphates were prepared using stoichiometric molar proportions. The solutions were mixed together and allowed to settle for 24 hours and the initial pH of the solution was measured. Two molar NaOH solutions was prepared and slowly added to the mixed solution of sulphates. H_2O_2 was also added to the mixed solution to increase the oxidation reaction. The mixed solution was constantly stirred and heated at low temperature (600C) during the addition of NaOH and H_2O_2.NaOH was added until the precipitation of dark brownish colour is formed and pH of the precipitation was measured. The precipitation was washed several times by acetone and then by double distilled water several times. The solution was filtered to get fine particles of Mg-Cr spinel ferrite system. The fine powder of Mg-Cr spinel ferrite system is then heated at 1500C for four hours to remove water molecules. The fine powders were sintered at 8000C for 12 hours. All the synthesized powders were characterized by using X-ray diffraction technique. Microstructure and surface morphology of all the samples were studied using Scanning Electron Microscope (JEOL JSM 840)

RESULT AND DISCUSSION

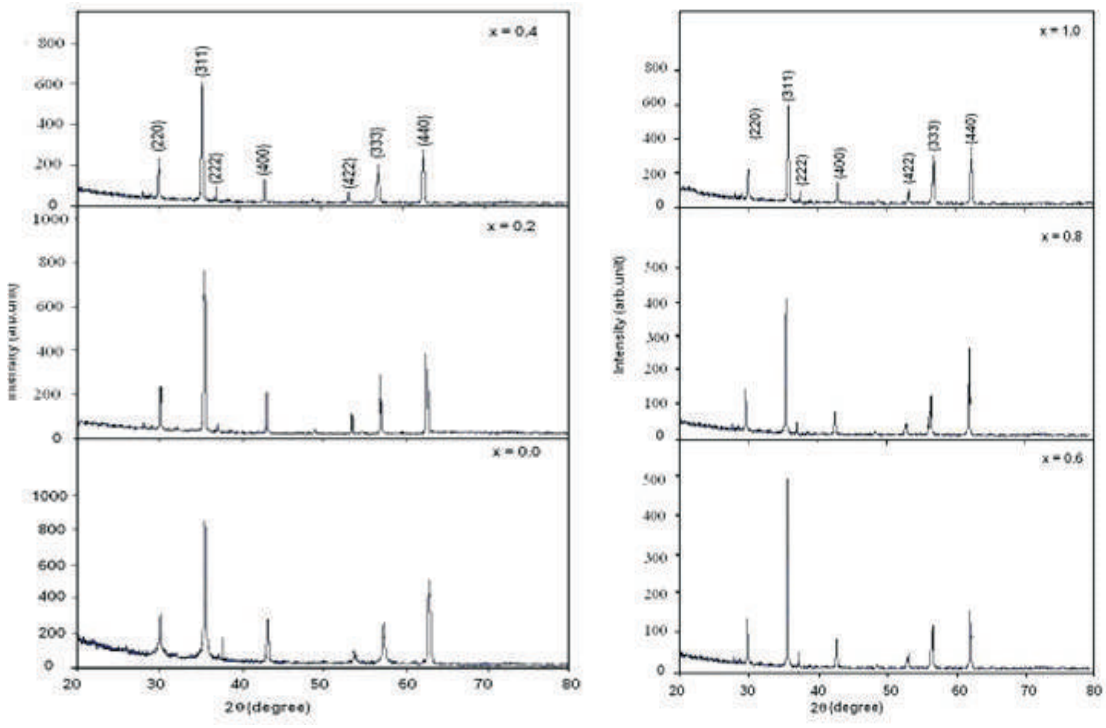


Fig. 1: Typical X-ray diffraction pattern for $MgFe_{2-x}CrO_4$ (x = 0.0, 0.2, 0.4, 0.6, 0.8 and 1.0)

X-ray diffraction patterns of all the composition of MgFe2-xCrxO4 spinel ferrite system are presented in Fig.1 The close examination of XRD patterns confirms the formation of single phase cubic spinel ferrites. The Millers indices (h k l) are assigned to each Bragg peak of the XRD pattern. The XRD pattern includes the planes (220), (311), , (222), (400), (422), (333), (440). corresponding to face centre cubic (FCC) spinel structure [6]. The values of inter planner spacing `d` were calculated using Braggs` law

and are presented in Table 5.1. This table also contains the corresponding Bragg angle values for all the compositions under investigation. The lattice parameter 'a' was calculated using the equation,

$$a \lambda d\sqrt{(h^2 \lambda k^2 \lambda l^2)}$$

where, d is the inter-planer spacing and (hkl) is the index of the XRD reflection peak. For an accurate calculation of lattice constant 'a' lattice parameter for each peak of XRD pattern was calculated and then taking average of them. The lattice parameter has been determined using XRD data with an accuracy of +0.002Å. The values of lattice parameter are given in Table 5.2. The variation of lattice parameter with chromium composition x is shown in Fig.1. The lattice parameter initially increases up to x = 0.4 and then decreases for further increase in Cr content x. The non-linear behaviour of lattice constant may be due to the fact that the spinel system under investigation (Mg-Cr) is not completely normal or inverse. Our results on lattice parameter are well supported by literature report [7]. The behaviour of lattice parameter with x of the present system is similar to that of ceramically prepared Mg-Cr system [8]. However, the values of lattice constant for the same compositions Mg-Cr system are different for wet chemically prepared system and ceramically prepared system.

The X-ray density 'dx' of all the samples was also calculated using the values of molecular weight and volume of unit cell parameter. Table 1 gives the values of X-ray density 'dx'. It can be seen from Table 1 that X-ray density increases with increase in chromium composition (x). The dependence of X-ray density with Cr content x is shown in fig 1. The particle size was determined using Scherrer formula [9] and by assuming peak width of highest intensity plane (311).

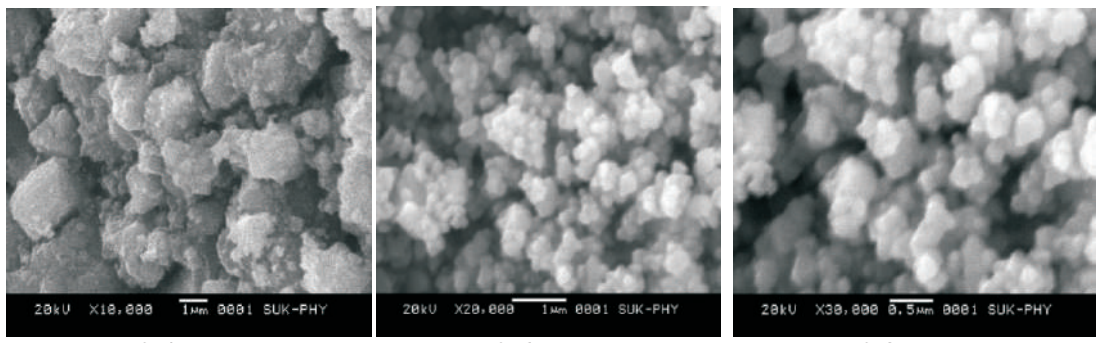
Table 1 Lattice constant (a), X-ray density (dx), Particle size (t) of the system MgFe (2-x) Cr(x) O_4 System

Composition(x)	Lattice constant(a)(A^o)		X-ray density(dx) gm/cm^3	Particle size (t) (nm)	
	Obs.	Cal.		XRD	SEM
Mg Fe_2 O_4	8.3741	8.3846	4.4205	42.87	51.11
Mg $Fe_{(1.8)}$ $Cr_{(0.2)}$ O_4	8.2961	8.3782	4.4481	34.54	44.23
Mg $Fe_{(1.6)}$ $Cr_{(0.4)}$ O_4	8.46	8.3894	4.4794	48.86	57..15
Mg $Fe_{(1.4)}$ $Cr_{(0.6)}$ O_4	8.451	8.3695	4.5073	45.69	49.97
Mg $Fe_{(1.2)}$ $Cr_{(0.8)}$ O_4	8.442	8.3541	4.5333	26.068	35.16
Mg FeCr O_4	8.431	8.3577	4.557	36.036	41.37

The XRD line width and particle size are calculated through the Scherrer equation

$$t \lambda \frac{0.9\lambda}{B\cos\lambda_B} \qquad (1)$$

Where, t is diameter of crystal particle, is wavelength of the X-ray radiation, B is Bragg's angle; B is measure of broadening of diffraction due to size effect. The estimated values of particle size for all the composition x are listed in Table 1 The particle size obtained from XRD data is of the order of few ten nano meters. The particle size was also obtained from scanning electron microscopy (SEM). Fig.2 gives the SEM images of typical samples (x = 0.4, 0.6 and 0.8). SEM images shows grains of irregular shape and no agglomeration is seen. These SEM images are used to estimate the particle size of the samples. The values of particle size obtained from SEM images are presented in table 1 It is observed from table 1 that the particle size of the presently investigated samples obtained from SEM images is in between 30 to 50 nm.



x=0.4 x=0.6 x=0.8

Fig : 2 Scanning electron micrograph for the typical samples of the system MgxFe(2-x)CrO (X = 0.4, 0.6, 0.8)

CATION DISTRIBUTION:

In order to determine the distribution of cations over the available tetrahedral (A) and octahedral [B] sites in MgFe$_{2-x}$Cr$_x$O$_4$ spinel ferrite system X-ray diffraction method has been used. In this method X-ray Intensities of various planes were calculated using the formula suggested by Burger [10],

$$Ihkl = |Fhkl|LP \cdot P \qquad (2)$$

Where, notations have the same usual meaning. The values of multiplicity factor P and Lorentz polarization factor are taken from the literature [9]. The structure factor F for various planes have been calculated and the values of structure F alongwith multiplicity factor P, and Lorentz polarization factor LP According to Ohnishi and Terenishi [11], the intensity ratio of planes (220), (400), (422) and (440) are considered to be sensitive to the cation distribution.

Table 2: Cation distribution and x-rays intensity ratio of Mg Fe (2-x) Cr(x) O_4

x	A-site	B-site	I(400)/I(222)		I(422)/I(511)	
			I cal	I obs	I cal	I obs
0	Mg(0.1) Fe(0.9)	Mg(0.9) Cr(0.0) Fe(1.1)	1.98	0.44	1.23	0.45
0.2	Mg(0.2) Fe(0.8)	Mg(0.8) Cr(0.2) Fe(1.0)	1.99	0.37	1.22	0.37
0.4	Mg(0.3) Fe(0.7)	Mg(0.7) Cr(0.4) Fe(0.9)	1.98	0.35	1.22	0.43
0.6	Mg(0.4) Fe(0.6)	Mg(0.6) Cr(0.6) Fe(0.8)	1.00	0.43	0.16	0.39
0.8	Mg(0.5) Fe(0.5)	Mg(0.5) Cr(0.8) Fe(0.7)	0.8	0.38	0.14	0.39
1	Mg(0.6) Fe(0.4)	Mg(0.4) Cr(1.0) Fe(0.6)	0.6	0.27	0.12	0.33

The X-ray intensity for the planes (220), (400), (422) and (440) has been calculated. The calculated intensity ratios for various possible distributions of cations (Mg^{2+}, Fe^{3+}, Cr^{3+}) over the tetrahedral (A) site and octahedral [B] site was then compared with the observed intensity ratio. A close agreement between calculated intensity ratios and observed intensity ratios, for a given distribution of cations gives

the correct cation distribution of the $MgFe_{2-x}Cr_xO_4$ spinel ferrite system. The cation distribution obtained from X-ray intensity ratio calculations is given in Table 2.

CONCLUSION

Wet chemical co-precipitation method plays an important role in governing the properties of the ferrite system. The nano particles of $Mg_{1-x}Zn_xFe_2O_4$ have been obtained successfully by wet chemical co-precipitation method. It is found that the lattice constants increase with increasing Cr content x. The nanosize nature of the particles was confirmed from the values of particle size obtained from XRD and SEM data. XRD analysis proves the formation of single phase cubic spinel structure. Cation distribution data shows that Cr^{3+} occupies octahedral B site where as Mg^{2+} and Fe^{3+} occupies tetrahedral A site.

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