



# X-Ray Diffraction and Structural Study of $\text{Cu}_{0.7+x}\text{Cd}_{0.3}\text{Zr}_x\text{Fe}_{2-2x}\text{O}_4$ ( $x = 0.1$ ) Mixed Spinel System

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**Abstract:** The crystal structure and lattice parameters of the Cu and Fe co-substituted disordered spinel series  $\text{Cu}_{0.7+x}\text{Cd}_{0.3}\text{Zr}_x\text{Fe}_{2-2x}\text{O}_4$  are investigated by means of X-ray diffraction (XRD) at room temperature (300K) and SEM results. The crystalline ferrite has been prepared by a modified Sol-Gel method. The analysis of XRD pattern suggests that the prepared  $\text{Cu}_{0.7+x}\text{Cd}_{0.3}\text{Zr}_x\text{Fe}_{2-2x}\text{O}_4$  ( $x = 0.1$ ) spinel ferrite possesses single phase cubic spinel structure. The intense peaks of the XRD pattern were used to determine the average crystalline size. The crystalline size obtained from XRD data using Scherrer formula is of the order of  $\sim 0.5\text{nm}$ . So far no one has reported any crystalline size smaller than 0.5 nm. The calculations from SEM results agreed within the limits. In present work the lattice parameter, particle size, lattice strain and studied with the different concentrations ( $x = 0.1$  to 0.4). The results were compared with ceramically prepared samples.

**Keywords:** X-ray diffraction, lattice constant, Sol-gel technique; Nanoparticle; SEM

## I. INTRODUCTION

The nickel ferrite ( $\text{CuFe}_2\text{O}_4$ ) is an inverse spinel taken to be collinear ferrimagnet.<sup>1,2</sup> The addition of trivalent ions like  $\text{Cd}^{3+}$  and  $\text{Zr}^{3+}$  in  $\text{CuFe}_2\text{O}_4$  influences the electrical and magnetic properties of the system.<sup>2-9</sup>  $\text{CuCr}_2\text{O}_4$  is normal spinel with canted ferromagnetic structure (canting of individual moments at octahedral (B) site)<sup>7-12</sup> having Curie temperature of 60 K.  $\text{CuCd}_2\text{O}_4$  is a partially inverted spinel in which the ratio of  $\text{Al}^{3+}$  ions in the tetrahedral (A) and octahedral (B) sites<sup>3</sup> is about 2:3.

The purpose of this paper is to prepare the mixed spinel  $\text{Cu}_{0.7+x}\text{Cd}_{0.3}\text{Zr}_x\text{Fe}_{2-2x}\text{O}_4$  ( $x = 0.1$  to 0.4) by co-substituting  $\text{Cd}^{3+}$  (non-magnetic) and  $\text{Zr}^{3+}$  (magnetic) ions in the ratio 2:1 ratio for  $\text{Fe}^{3+}$  in  $\text{CuFe}_2\text{O}_4$  and to investigate this system with a view to determine the effect of changing Fe: Cd: Zr ratio on the structural properties by X-ray diffraction and SEM measurements.

## II. EXPERIMENTAL SECTION

Four samples of Cd and Zr co-substituted  $\text{Cu}_{0.7+x}\text{Cd}_{0.3}\text{Zr}_x\text{Fe}_{2-2x}\text{O}_4$  ( $x = 0.1$  to 0.4) series were prepared by the sol-gel process for  $x=0.1$  to 0.6 in the steps of 0.1. The starting materials were  $\text{Fe}_2\text{O}_3$ ,  $\text{Cu}_2\text{O}_3$ ,  $\text{Cd}_2\text{O}_3$  and  $\text{Zr}_2\text{O}_3$ , all 99.9% pure supplied by Badar Chemicals Ltd. The oxides were mixed thoroughly in stoichiometric proportions to yield the desired composition then dry grounded. The mixture is mixed with PAA solution to prepare sol-gel of the mixture. The mixture is then heated in a furnace for 3hr. and then annealed. The resulting mixture is then powdered and pressed into pellets. The X-ray pattern of all the samples were recorded at room temperature with a Philips diffractometer using Cu-K $\alpha$  radiation and diffractometer showed sharp lines corresponding to single phase spinel.

## III. RESULTS AND DISCUSSION

The values of the lattice constant 'a' determined from X-ray data analysis with an accuracy of  $\pm 0.002 \text{ \AA}$ , of the series  $\text{Cu}_{0.7+x}\text{Cd}_{0.3}\text{Zr}_x\text{Fe}_{2-2x}\text{O}_4$  ( $x = 0.1$  to 0.4) plotted as shown is as shown in the Fig. 1. As per variation of 'a' vs. x, it is found that 'a' decreases with x slowly up to  $x = 0.3$  and thereafter it increases with x from  $x=0.3$  to  $x=0.4$  nearly obeying



Vegard’s law. Comparing present results with ceramically prepared sample as shown in Fig. 2. it decreases linearly from x=0.1 to 0.3 and thereafter it decreases non linearity from x = 0.4 onwards. Interestingly though the results are comparable up to x = 0.3 but, thereafter due to nanosized particles and ionic radius ‘a’ is increasing linearly with x from x = 0.3 onwards obeying Vegard’s law.<sup>13-15</sup>

Table 1: Lattice parameter and Density of the series  $Cu_{0.7+x}Cd_{0.3}Zr_xFe_{2-2x}O_4$  (x = 0.1 to 0.4)

Concentration ‘x’	Lattice parameter ‘a’ in A.U.	Density ‘rho’ $cm^{-3}$
0.1	7.4214	12.45
0.2	6.5152	11.20
0.3	5.5813	14.21
0.4	6.3244	10.51

Because of nano size particles the X-ray density of the sample show nonlinear behavior from x = 0.1 to x=0.3 and thereafter it displays linear decrease with x = 0.3. Ceramically prepared samples display linear slow decrease with x = 0.1 to x = 0.4, whereas nanosized samples show entirely different behavior due to nanosized of the particles.

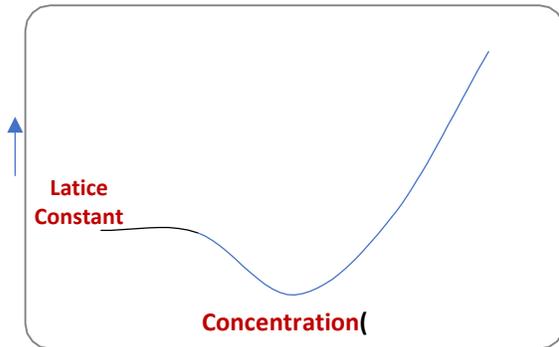


Fig. 1: Variation of the lattice parameter(a) with Cd-Zr concentration (X)

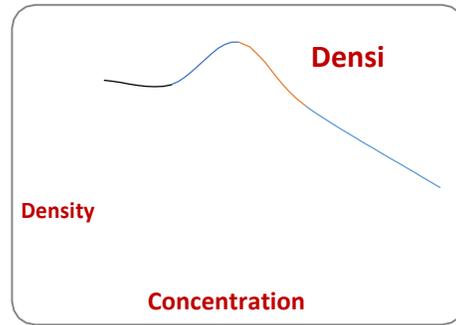


Fig. 2: Variation of the density(ρ) with Cd-Zr concentration (X)

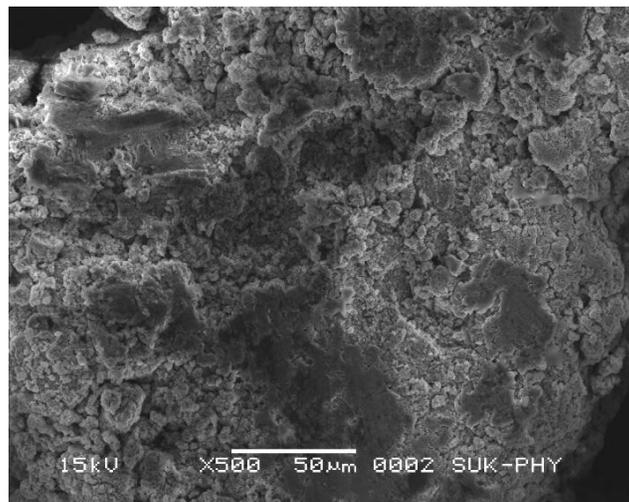


Fig. 3: SEM depicting the nanostructure of pellet of x = 0.1.

SEM was used to study morphological variations. The observed nanostructure from SEM image of x = 0.1 samples is depicted in Fig. 3. indicates that Cd and Zr co substitution for Fe in  $Cu_{0.7+x}Cd_{0.3}Zr_xFe_{2-2x}O_4$  (x = 0.1). These results are observed with particles.

#### **IV. CONCLUSION**

The values of lattice constant 'a' and X-ray density from X-ray data are given table 1. An interesting result due to nanosized of the particles are showing in lattice constant and X-ray density has been observed from the figure.

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