

STRUCTURE AND CATION DISTRIBUTION OF Cd-Zr CO-SUBSTITUTED NICKEL SPINEL FERRITE

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ABSTRACT

Cd-Zr co-substituted nickel spinel ferrite was prepared using standard ceramic method of preparation. The prepared samples were characterized by XRD for its structural analysis. The lattice parameter, particle size and X-ray density were obtained from the XRD data. Further, using XRD data the cation distribution was obtained revealing that the Zr^{4+} ions occupy both tetrahedral and octahedral sites.

Keywords: Cation Distribution, XRD Pattern, Lattice Parameter, Ceramic Method, X-Ray density.

INTRODUCTION

Spinel ferrites are widely used in many electronic devices. These are preferred because of their high electrical resistivity, chemical stability, mechanical hardness and reasonable cost [1-4]. These are also useful to prevent and eliminate radio frequency interference to audio systems. The systems made up of nano-particles are intensively studied both theoretically and practically due to their electric, dielectric and magnetic properties that are sensibly different from those of the bulk materials and their possible applications in various fields.

Ni-Cd Ferrites are well-known technological magnetic materials finding their applications in various electronic devices. The physical properties of the Ni-Cd ferrite are very sensitive to the method of preparation, the amount and the type of substitutions [5]. The basic condition process for Ni-Cd ferrite is due the thermally activated hopping of electron from one cation to another [6] and not due to thermal creation of charge carriers.

Ni-Cd ferrite is generally used at high frequency due to its low losses: the losses in these materials are also influenced by the grain size. Cadmium is known to play a decisive role in determining the ferrite properties [7] cadmium is responsible for the modification of ferrite properties. Nickel and cadmium are known to have very strong preference for the tetrahedral and octahedral sites, respectively, making Ni-ferrite a model inverse ferrite and Cd-ferrite a model normal ferrite [8]. However, nano-sized Ni- and Cd- ferrite are known to exist as mixed spinel structures [9, 10]. However, the synthesis route also plays a crucial role, so that samples of comparable crystallite size prepared by different processes show different electrical and magnetic properties. This paper illustrates structural and cation distribution studies of co-substituted Cd-Zr nickel spinel ferrite.

EXPERIMENTAL

PREPARATION METHOD

Ni-Cd ferrite of composition $Ni_{0.7+x}Cd_{0.3}Zr_xRe_{2-2x}O_4$ where $x=0.00$ and 0.3 were prepared by double sintering ceramic technique, using A.R. grade Nickel Oxide (NiO), Cadmium Oxide (CdO), Zirconium dioxide (ZrO_2) and Ferric oxide ($Fe^{2+}O_3$) compounds with 99.9% purity. They were mixed in their stoichiometric proportion, milled with acetone base agate mortar for about three hours to get fine powder. The pre-sintering was carried out at $500^\circ C$ for 24 hours then furnace is switched off; samples were allowed to cool in furnace by natural process. These sintered powders were then pressed into pellets of desired thickness with the help of hydraulic press by applying a

pressure of 6 tones for 10 minutes on each pellet. Further these pellets were finally sintered at $1100^\circ C$ for 24 hours and cooled in natural process.

CHARACTERIZATIONS

The powdered X-ray diffraction patterns of all the samples were recorded at room temperature using Cu-K α radiation on Philips (PW 3710) X-ray diffractometer. X-ray diffraction data was collected in the 2θ range of 20° to 80° with scanning rate $2^\circ/\text{minute}^{-1}$. The X-ray diffraction pattern shows sharp Bragg peaks corresponding to single phase spinel structure. The XRD data have been used to determine the values of lattice parameter, X-ray density, and particle size and cation distribution.

RESULTS AND DISCUSSION

X-ray diffraction

X-ray diffraction patterns of the samples are recorded at room temperature represented in Fig 1. these figures show well defined peaks and (311) reflection appears to be more intense in both the samples. All the planes are allowed planes which confirm the formation of single phase cubic spinel structure without appearance of any extra peaks.

The Inter- planer spacing (d) values were calculated for the recorded peaks using Bragg's law and the lattice constant 'a' was calculated for each plane using the relation.

$$a = d \sqrt{h^2 + k^2 + l^2}$$

The average lattice constant 'a' for each sample was calculated and presented in table 1.

It is found that the lattice constant increase with Zr^{4+} ion substitution. In the present system, the increase in a lattice constant is attributed to replacement of smaller ionic radius of Fe^{3+} (0.67\AA) by larger ionic radius Zr^{4+} (0.80\AA) obeying Vegard's Law [11, 12]. The results of increase of lattice constant with Zr^{4+} substitution are in good agreement with other Zr^{4+} substituted spinel ferrite [13, 14].

The particle size is determined from XRD data with full width at half maxima using Debye-Scherrer formula. The Values of particle size are listed in Table 1. The X-ray density (d_x) was determined using the relation.

$$d_x = \frac{Zm}{N_A a^3}$$

The variation of d_x is shown in table 1. It is clear from table 1 that the increase in X-ray density is because of the increase in mass over takes the increase in volume of the unit cell.

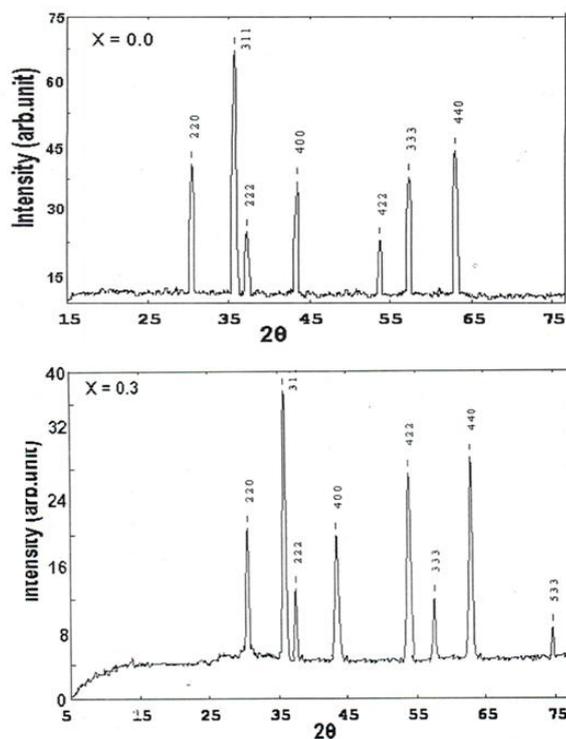


Fig.1: XRD Patterns of $Ni_{0.7+x}Cd_{0.3}Zr_xFe_{2-2x}O_4$ ($x = 0.0$ and 0.3)

Table 1: Lattice constant (a), X-ray density (d_x), and particle size (t) of $Ni_{0.7+x}Cd_{0.3}Zr_xFe_{2-2x}O_4$ ($x=0.00$ and 0.3)

Comp. X	Lattice constant (Å)	X-ray density (gm/cm ³)	Particle size (Å)
0.0	8.315	5.785	206.0
0.3	8.345	5.983	203.9

Cation distribution

The cation distribution was calculated using R-factor method. In this method the best structure is selected so that the value of residual function R is minimized. The intensities of planes which are mostly sensitive to cations on tetrahedral and octahedral sites are taken in to consideration while determining the cation distribution in the presently selected system.

In cation distribution Ni^{2+} and tiny part of Cd^{2+} are occupied at B-site [15] and major part of Cd^{2+} is occupied at (A)-site. For Zr^{4+} -ion distribution, it was reported that Zr^{4+} ions first enter the (A) sites for low concentration and then subdivide between (A) and (B) site for higher concentration [16]. Hence Zr^{4+} and Fe^{3+} occupied at both (A) and (B) site. The cation distribution of the present ferrite samples is listed in Table 2. From table 2 it is clear that the calculated and observed intensity ratios are in good agreement to each other.

CONCLUSION

X-ray diffraction patterns indicate that the samples possess single phase cubic spinel structure. The lattice constant obtained from XRD data increases with the increase in Zr^{4+} content x. In cation distribution Ni^{2+} and part of Cd^{2+} are occupied at B-site and major part of Cd^{2+} is occupied at A-site. Zr^{4+} and Fe^{3+} is occupied at both (A) and (B) site.

Table 2: Cation distribution and intensity ratio of $Ni_{0.7+x}Cd_{0.3}Zr_xFe_{2-2x}O_4$ ($x=0.00$ and 0.3)

Comp. X	Cation distribution		Intensity ratio $I_{(400)}/I_{(440)}$	
	A-Site	B-site	Obs.	Cal
0.0	($Cd_{0.24}Fe_{0.76}$)	($Ni_{0.7}Cd_{0.06}Fe_{1.24}$)	0.847	1.165
0.3	($Cd_{0.27}Zr_{0.2}Fe_{0.53}$)	($Ni_{1.0}Cd_{0.03}Zr_{0.1}Fe_{0.87}$)	0.714	1.22

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