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REVIEW OF RESEARCH

SOL-GEL SYNTHESIS AND XRD INVESTIGATIONS OF CR^{3+} DOPED NCZ-FERRITE NANOPARTICLES

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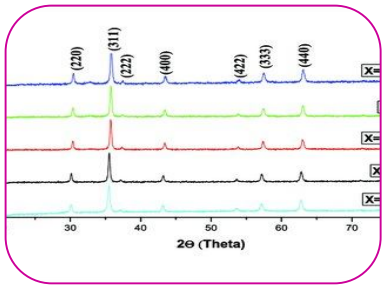
ABSTRACT



Sol-gel synthesized NCZ-ferrite nanoparticles doped by Cr^{3+} ions have the general chemical formula $Ni_{0.2}Co_{0.6}Zn_{0.2}Fe_{2-x}Cr_xO_4$ with x = 0.0 to 1.0 in the step of 0.2. All the prepared samples were characterized by X-ray diffraction technique. The analysis of X-ray diffraction patterns indicates that the samples possess single phase cubic spinel structure. The X-ray diffraction data is used to obtain the structural parameters viz. lattice parameter, X-ray density and particle size. The values lattice parameter is found in the range 8.36 to 8.29 in decreasing order with Cr^{3+} concentration increases in the composition. This behavior of lattice parameter is related to the ionic radii of constituent ions. In the present investigation Fe^{3+} ions with larger ionic radii (0.67Å) are replaced by Cr^{3+} ions with relatively small ionic radii (0.63Å) which decreases the lattice parameter. Average particle sizes of all the samples are obtained in nanometer range and shows decreasing trend with Cr content 'x'. The X-ray density, Bulk density, percentage porosity and specific surface area were also calculated.

KEYWORDS: *Sol-Gel technique, Lattice Parameter, Particle size.*

INTRODUCTION:

In recent years, several synthesis methods have been reported to prepare nanosized pure and doped ferrites [1-4]. Some of these methods need high sintering temperature, high cost and long time for preparation. Among these methods, sol-gel auto-combustion method is the promising method to prepare fine particles of ferrite [5]. This method is one of the methods of chemical synthesis which requires low temperature and cost effective. In this method mixing was made at molecular level which gives good homogeneity and controls the cation distribution. Nanostructured materials show unique properties and completely different from bulk materials. Due to extremely small dimensions maximum atoms present at surface and the surface to volume ratio increases which changes the structural, magnetic and electric properties of the materials than the bulk materials.

Ferries are used in wide range of a.c. technological applications, especially in core transformers, recorder head, etc. [6] due to their magnetic and electrical properties. Polycrystalline soft ferrites are well known dielectric material and are useful in microwave application, high quality filter rod antennas, radio frequency circuits, transformer core, sensors etc. [7-10]. Amongst all the ferrites, copper ferrite is unique in the contest of establishing mutual relationship between the magnetic and structural properties. In the past, the magnetic behaviour of copper mixed ferrite, doped with diamagnetic ions such as Zn^{2+} [11], Al^{3+} [12],

Ge^{4+} [13], and Ti^{4+} [14] have been subject to various studies because of their technological application. It has been reported [15] that, depending on the type and concentration of diamagnetic dopant specific physical properties can be achieved. NCZ-ferrites are technologically important materials as they are having unique physical and magnetic properties. Particularly cobalt ferrite has high coercivity and low loss of eddy currents [5]. The Nickel –Zinc ferrites are extensively used in high frequency microwave devices for low coercivity and high resistivity [16]. In the present paper we have reported the sol-gel synthesis and structural parameters of Cr^{3+} doped NCZ ferrites.

EXPERIMENTAL:

Synthesis:

Sigma-adrich made 98.5% pure nitrates of constituent elements such as Nickel Nitrate ($Ni(NO_3)_2 \cdot 6H_2O$), Cobalt Nitrate ($Co(NO_3)_2 \cdot 6H_2O$), Zinc Nitrate ($Zn(NO_3)_2 \cdot 6H_2O$), Chromium Nitrate ($Cr(NO_3)_3 \cdot 9H_2O$), Ferric Nitrate ($Fe(NO_3)_3 \cdot 9H_2O$) were used as starting materials to prepare the $Ni_{0.2}Co_{0.6}Zn_{0.2}Fe_{2-x}Cr_xO_4$ with x = 0.0 to 1.0 in the step of 0.2. All the nitrates of constituent elements with their weight proportion were mixed thoroughly with distilled water in which citric acid was added as a chelating agent in the weight proportion 1:3. The whole mixture is subject to continually stirring at constant temperature 90^0C. Liquid ammonia was poured in the mixture continuously to maintain the pH=7 of the solution. After nearly 2 hours the solution is converted into viscous sol and thereafter by continuous heating and stirring it converted into dried Gel. After self ignition of dried gel the compound converted into fine particles of ferrite. The self ignited powders are sintered at 600^0C for 4hrs and grinded to obtain the fine, pure and homogeneous ferrite particles. The final products are then characterized by X-ray diffraction technique in order to study the structural properties.

Characterization:

All the samples of $Ni_{0.2}Co_{0.6}Zn_{0.2}Fe_{2-x}Cr_xO_4$ with x = 0.0 to 1.0 in the step of 0.2 were characterized by X-ray diffraction (XRD) technique on Rigaku difractometer having Cu-K_α radiation (λ=1.5404 Å). The XRD data was collected at room temperature in the 2θ range 20^0 to 80^0. The scanning rate was maintained at 2^0/min. By using the XRD data, the structural parameters like lattice constant, X-ray density, particle size etc.

RESULTS AND DISCUSSION:

Sol-gel synthesized $Ni_{0.2}Co_{0.6}Zn_{0.2}Fe_{2-x}Cr_xO_4$ ferrite powders were characterized by X-ray diffraction technique at room temperature. Fig. 1 shows the X-ray diffraction patterns of typical samples for x = 0.0, 0.4 and 0.8. All the Bragg's peaks are appear sharp and no impurity peaks are obtained. The Bragg's reflections are obtained for the planes 220, 311, 222, 400, 422, 333, 440. All the characteristics peaks of Ni-Co-Zn-Cr ferrites are present in the XRD patterns. The peak intensity decreases with Cr substitution in Ni-Co-Zn ferrite and the Bragg's angles were shifted slightly towards higher angles. The broadening of peaks increases gradually with increase in Cr concentration. This clearly indicates that the particle size decreases with Cr content 'x' increases in composition.

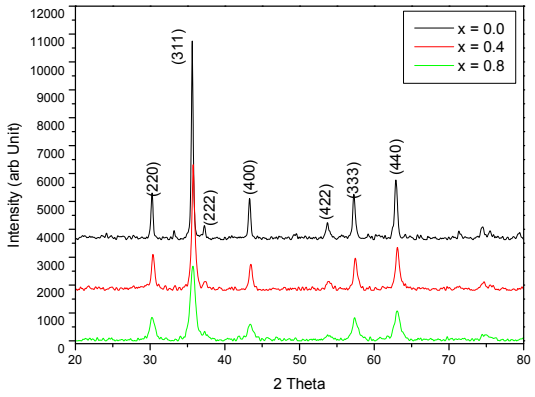


Fig. 1: XRD patterns of $Ni_{0.2}Co_{0.6}Zn_{0.2}Fe_{2-x}Cr_xO_4$ for x = 0.0, 0.4, 0.8.

The lattice parameter of all the samples was calculated by using the following relation [17],

$$a = d\sqrt{\left(h^2 + k^2 + l^2\right)} \qquad (1)$$

where, d is the interplanner spacing and (hkl) is the miller indices of the XRD reflection peaks. The calculated values of lattice parameter 'a' are listed in Table 1 and the variation is shown in Fig. 2. From fig. 2, it is clear that lattice constant 'a' decreases with increasing Cr^{3+} content in NCZ-composition. The increase in lattice constant is explained on the basis of difference in ionic radii of Cr^{3+} ions and Fe^{3+} ions. In the present investigation smaller Cr^{3+} ions (0.63Å) replaces the larger Fe^{3+} ions (0.67Å). The increase in lattice constant and shifting of peaks towards higher angles with Cr^{+3} concentration reveals that the Cr atoms have substituted into the composition homogeneously.

Table 1: Lattice parameter 'a', X-ray density 'd_X', Bulk Density 'd_B', percentage porosity 'P', particle size 't' and specific surface area 'S' of the series $Ni_{0.2}Co_{0.6}Zn_{0.2}Fe_{2-x}Cr_xO_4$.

'x'	Lattice Constant 'a' (Å)	X-ray density 'd_X' (g/cc)	Bulk Density 'd_B' (g/cc)	Percentage porosity 'P' (%)	Particle Size 't' (nm)	Specific Surface area 'S'
0.0	8.3532	5.376	4.051	24.63	33.53	44.18
0.2	8.3496	5.365	4.171	22.26	30.74	46.80
0.4	8.3398	5.366	4.215	21.46	28.36	50.20
0.6	8.3247	5.377	4.292	20.18	26.55	52.66
0.8	8.3155	5.378	4.337	19.36	24.44	56.62
1.0	8.2951	5.399	4.396	18.58	22.90	59.59

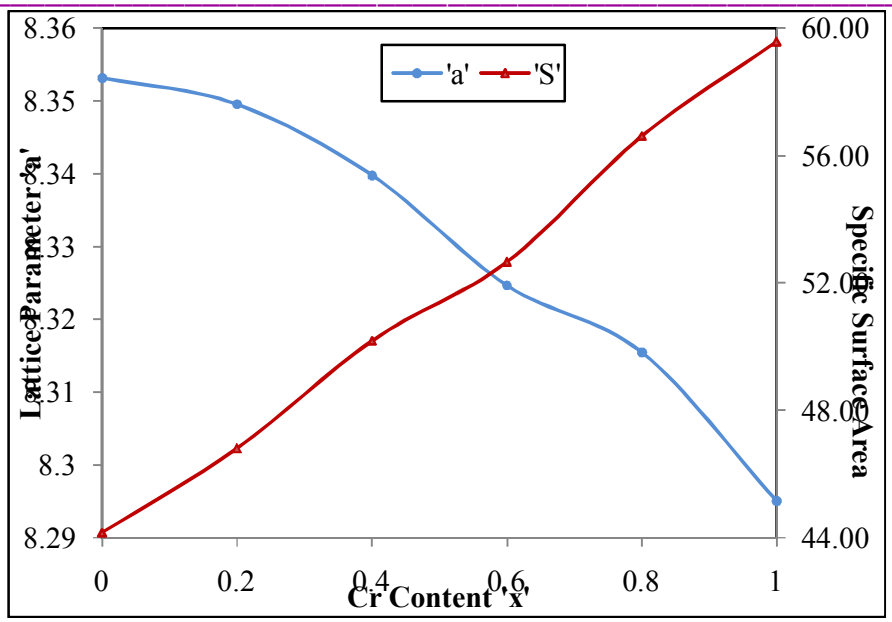


Fig. 2: Variation of lattice constant 'a' and Specific surface are 'S' for $Ni_{0.2}Co_{0.6}Zn_{0.2}Fe_{2-x}Cr_xO_4$.

The X-ray density 'd_X' was calculated for all the samples by suing molecular weight of the sample and lattice parameter. The calculated values of X-ray density are given in Table 1. The bulk density for all the samples was calculated by using standard mass – volume ratio and the values are listed in table 1. Bulk density found increases as the Cr percentage increases. By using the values of X-ray density and bulk density, percentage porosity was calculated by using following realtion [18],

$$P = \left(\frac{d_X - d_B}{d_X} \right) \times 100 \qquad (2)$$

where, d_X and d_B are the X-ray and bulk densities respectively. The calculated values of percentage porosity are shown in table 1. It is clear from table 1 that the porosity (P) decreases with increase in Cr content 'x'. The decrease in porosity is due to increase in bulk density. The broadening in XRD peaks indicates the particles are in nano scale. In the present investigation the particle size was calculated by using most intense peak (311). Following Scherrer's formula is used to calculate the particle size [18],

$$t = \frac{0.9\lambda}{B\text{Cos}\theta_B} \qquad (3)$$

Where, 'B' is FWHM in (2θ), θ_B is the corresponding Bragg's angle and λ is wavelength of incident radiation. Particle size calculated by using the relation (3) lies in the range 34nm to 22nm. The decrease in particle size with increase in Cr content also led to increase in specific surface area (S). The specific surface area was calculated by using the following equation [19];

$$S = \frac{6000}{td_B} \qquad (4)$$

where,'t' is particle size and d_B is the bulk density. The calculate values of specific surface area 'S' are given in Table 1 and the variation is shown in Fig. 2. The increase in specific surface area is due to decrease in crystalline size.

CONCLUSIONS:

Sol-gel auto combustion technique successfully prepares the nanocrystalline Cr^{3+} ion substituted Ni-Co-Zn ferrite powders. From X-ray diffraction data it is clear that, all the samples possess single phase cubic spinel structure. All the XRD peaks are sharp and no impurity peaks are observed which indicates that the prepared samples are pure and homogeneous. The lattice constant calculated from XRD data is increases with increase in Cr content which is clearly related with the difference in ionic radii of Cr^{3+} and Fe^{3+} ions. The particle size is obtained in nanometer range and varies in decreasing order with increase in Cr concentration. The specific surface area increases with increase in Cr content which is due to decrease in grain size.

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