

Composition Dependence of Elastic Moduli of Mixed Cobalt-Zinc Ferrites Prepared by Citrate Precursor Method

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$\text{Co}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ nanoparticles were prepared by chemical citrate precursor synthesis method. The Young's modulus 'E' and the rigidity modulus 'n' of mixed cobalt-zinc ferrites have been determined by the ultrasonic pulse transmission technique at 1 MHz. The elastic modulus of the ferrites were corrected to zero porosity using the formulae of Harselman and Fulrath. The observed variation of the elastic modulus with composition has been interpreted on the basis of binding forces between the atoms of the spinal lattice. A linear relationship between Debye temperature Θ_D and average sound velocity V_m has also been observed.

Keywords: $\text{Co}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ nanoparticles, Citrate precursor synthesis, Elastic properties.

INTRODUCTION

Nanoferrites is the emerging field of study from past years because of their fabulous applications in the areas electronic, magnetic and catalytic properties. Spinel ferrites have the permeability and high resistivity at high frequencies and specific crystallographic structure and microstructure as prove a huge range of applications. They have diverse magnetic properties as used to prepare many electromagnetic devices [1,2] such as information storage system, medical diagnostics, microwave devices [3] radar, digital recordings in catalysis, magnetic refrigeration, in ferrofluid technology [4] and in photo magnetism [5], magnetic drug delivery, hyperthermia [6], for cancer treatment [7] and magnetic resonance imaging [8]. Cobalt ferrite is one of the potential candidates for magnetic and magneto-optical recording media [9,10].

The extensively used chemical methods for the synthesis of ferrites are glyoxylate precursor method [11], sucrose method [12], reverse micelle technique [13], hydrothermal method. In citrate precursor method citric acid is the organic precursor that acts as multidentate ligand and complexes with multivalent atoms to form chelates. High uniformity of metallic constituents and a high degree of chelation of the metal ions increases with increasing the concentration of citric acid.

Citrate method gives the lowest value for the lattice parameter and particle size and yields more homogenous nanomaterials at lower processing temperatures. Thus, it presents a large superficial area and good sinterability in relation to powders obtained by other synthesis techniques [14-18].

The cation Co^{2+} in the cobalt-zinc ferrite promotes the exchange reaction $\text{Co}^{2+} + \text{Fe}^{3+} \rightleftharpoons \text{Co}^{3+} + \text{Fe}^{2+}$ in octahedral sites, whereas tetrahedral sites are preferentially occupied by zinc cations. The nature of the binding forces in the solid compounds was represented by the elastic constant and it reveals the thermal properties of solids so the elastic modulus explores the mechanical strength, fracture toughness and thermal shock resistance [19]. The widely used ultrasonic pulse transmission technique is the most common technique for elastic constants and Debye temperature determination [20]. On the basis of their extensive applications, it became a fascinating subject to study the composition dependence of the elastic moduli of Co-Zn ferrites. The outcome of such a study is reported in this publication.

EXPERIMENTAL

The chemical reagents used $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ were of analytical grade and procured from

Merck chemicals, Mumbai, India. Citric acid was purchased from SD fine chemicals Mumbai, India.

Synthesis of cobalt-zinc ferrites: The cobalt-zinc ferrites employed in this present investigation have the composition formula $\text{Co}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ (where $x = 1.0, 0.9, 0.8, 0.6, 0.4, 0.2$) were synthesized using citrate precursor method. The chemical reagents used were $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ and citric acid all the reagents were used without any further purification and were weighed out in the required stoichiometric proportion and dissolved separately in deionized water. The individual solutions were mixed together except ferric nitrate. The aqueous ferric nitrate solution is added drop by drop with continuous stirring to form a homogeneous solution under heat treatment, a high residue was formed and within a few hours a dried gel is formed finally. This dried gel is sintered at 600°C for 4 h, followed by slow cooling.

Characterization: The ultrasonic compressional V_1 and shear V_s velocities at room temperature were determined for all the mixed cobalt-zinc ferrites using an ultrasonic technique [21], altered and improved by the use of a digital oscilloscope (Tektronix 2230) and utilizing cursor movement for travel time measurement [22]. The synthesis of cobalt-zinc ferrites has been done using Metrax India limited make muffle furnace workable up to 1450°C .

RESULTS AND DISCUSSION

The lattice parameter for the mixed cobalt-zinc ferrites is reported in Table-1. In case of cobalt-zinc ferrites, the values incurred are in good accord with that reported by several workers [23-27]. The values of lattice parameter for compositions CoFe_2O_4 , $\text{Co}_{0.9}\text{Zn}_{0.1}\text{Fe}_2\text{O}_4$, $\text{Co}_{0.8}\text{Zn}_{0.2}\text{Fe}_2\text{O}_4$, $\text{Co}_{0.6}\text{Zn}_{0.4}\text{Fe}_2\text{O}_4$, $\text{Co}_{0.4}\text{Zn}_{0.6}\text{Fe}_2\text{O}_4$ and $\text{Co}_{0.2}\text{Zn}_{0.8}\text{Fe}_2\text{O}_4$ are represented in Fig. 1. Linearity has been noticed with an increase in cobalt concentration. So it is proven that the mixed cobalt-zinc ferrites obey the Vegard's law [28]. The relevant linear variation is observed in the case of nickel-zinc ferrites, aluminum substituted MgFe_2O_4 [29] and $\text{Zn}_x\text{F}_{3-x}\text{O}_4$ [30].

The cation distribution of cobalt-zinc ferrite, having an inverse spinel structure, and the composition is given by $\text{Fe}^{3+}[\text{Co}^{2+}\text{Fe}^{3+}]_2\text{O}_4$. We use the pattern that the parenthesis () and square brackets [] indicate ion disseminated on tetrahedral and

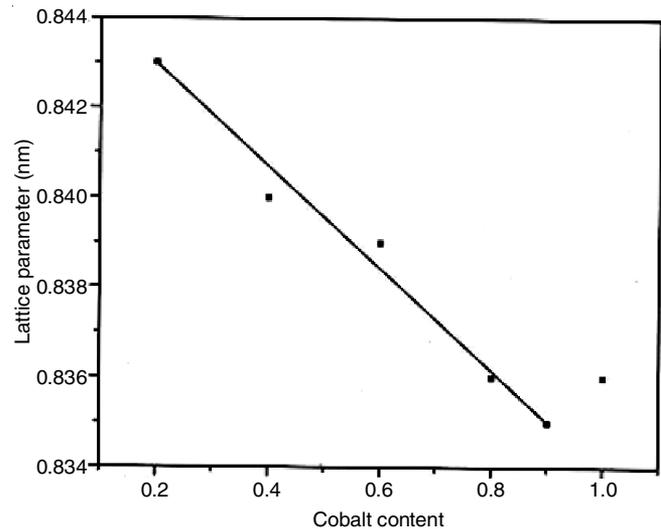


Fig. 1. Plot of lattice parameter with cobalt content

octahedral sites respectively. The non-magnetic cation M^{2+} have a larger radius than those of Fe^{3+} and Zn^{2+} cations can be substituted for Co^{2+} magnetic ions. Zn^{2+} ions invaded the tetrahedral sites [31] results in the substitution of Fe^{3+} ions to the empty octahedral sites left by Co^{2+} ions. The cation distribution of Co-Zn ferrites is $\text{Fe}_x^{3+}\text{M}_{1-x}^{2+}[\text{Co}_x^{2+}\text{Fe}_{1-x}^{3+}\text{Fe}^{3+}]_2\text{O}_4$. The relation between tetrahedral ionic radius and the lattice parameter can be deduced as follows. The given composition x , the mean ionic radius per molecule of tetrahedral site [32] may be defined as:

$$r_{\text{tetra ion}} = (1-x)r_{\text{tetra}}(\text{Fe}^{3+}) + Xr_{\text{tetra}}\text{Me}^{2+} \quad (1)$$

Here, Me^{2+} refers to the substituted divalent metal. The mean tetrahedral ionic radius for each composition has been reckoned by using the above relation. A plot of lattice parameter versus tetra ionic radius for Co-Zn ferrites is depicted in Fig. 2. The above similarity is noticed for Ni-Zn, Mn-Cd [33] and Li-Ni [34] ferrites.

The bulk density was determined exactly by the hydrostatic method while the X-ray density was reckoned from the obtained values of the lattice parameter using the formula [35].

$$\rho = \frac{8M}{N a^3} \quad (2)$$

TABLE-1
LATTICE PARAMETER AND TETRAHEDRAL IONIC RADIUS VALUES OF MIXED Co-Zn FERRITES AND DENSITY AND POROSITY DATA FOR MIXED Co-Zn FERRITES

Ferrite	Lattice parameter (nm)	Lattice parameter values from literature (nm)*	Tetrahedral ionic radius (nm)	Bulk density 10^3 Kg m^{-3}	X-ray density 10^3 Kg m^{-3}	Porosity (%)	Ref.
CoFe_2O_4	0.8367	0.835 0.838	0.049	5.01	5.37	5.6	[3] [4]
$\text{Co}_{0.9}\text{Zn}_{0.1}\text{Fe}_2\text{O}_4$	0.8354	0.837 0.833 0.839	0.051	5.09	5.36	5.04	[3] [5] [6]
$\text{Co}_{0.8}\text{Zn}_{0.2}\text{Fe}_2\text{O}_4$	0.836	0.837 0.838	0.054	5.11	5.36	4.66	[3] [6]
$\text{Co}_{0.6}\text{Zn}_{0.4}\text{Fe}_2\text{O}_4$	0.8392	0.841 0.841	0.059	5.12	5.33	3.94	[3] [4]
$\text{Co}_{0.4}\text{Zn}_{0.6}\text{Fe}_2\text{O}_4$	0.8408	0.841 0.842	0.064	5.14	5.32	3.38	[4] [4]
$\text{Co}_{0.2}\text{Zn}_{0.8}\text{Fe}_2\text{O}_4$	0.8435	0.846	0.069	5.18	5.3	2.26	[7]

*See reference shown in last column.

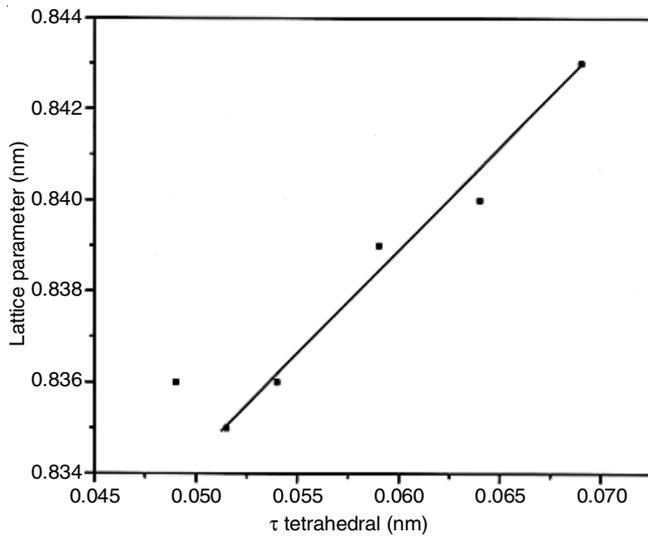


Fig. 2. Plot of lattice parameter and tetrahedral ionic radius

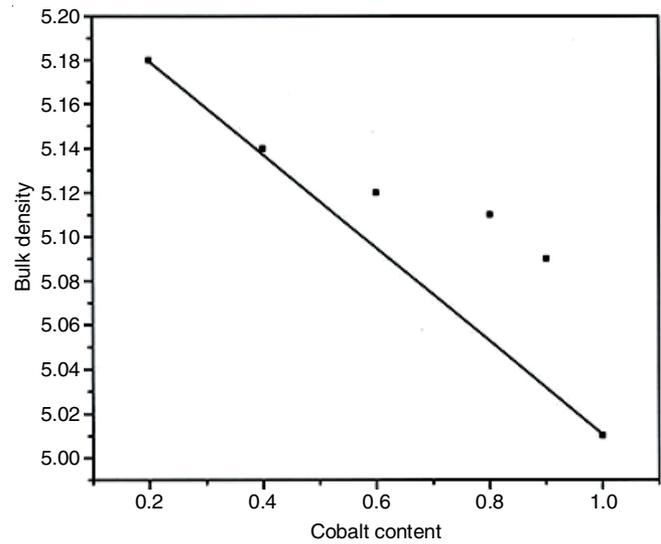


Fig. 3. Variation of bulk density with cobalt content

where 8 represents the number of molecules in a unit cell of the spinal lattice, ρ is the X-ray density, M is the molecular weight of the sample, N is the Avogadro's number and 'a' is the lattice parameter of the Co-Zn ferrites. The percentage porosity 'p' of the ferrite nanoparticles was determined by using the following formula

$$\text{Percentage porosity (p)} = \left(1 - \frac{\text{Bulk density}}{\text{X-ray density}}\right) \quad (3)$$

The values of bulk density, X-ray density and percentage porosity for the cobalt-zinc ferrites are given in Table-1 and the Fig. 3 indicates a plot of X-ray density *versus* Co content for the specimens.

The elastic moduli of the samples were measured using the following expressions:

$$[\text{Longitudinal modulus (L)} = \rho(V_1)^2] \quad (4)$$

$$\text{Rigidity modulus (n)} = \rho(V_s)^2 \quad (5)$$

$$\text{Bulk modulus (K)} = L - \frac{4}{3}n \quad (6)$$

$$\text{Young's modulus (E)} = (1 + \sigma)2n \quad (7)$$

$$\text{Poisson's ratio } (\sigma) = \frac{L - 2n}{2(L - n)} \quad (8)$$

where V_1 and V_s are the longitudinal and shear velocities and ρ is the density. The values of V_1 , V_s , E , n , K and σ for different concentrations of the Co-Zn ferrites are expressed in Table-2.

The ferrites were found to be porous with porosity varying from 2.26 to 5.60 %. Hence, the elastic moduli given in Table-2

have been corrected to zero porosity using the formulae of Hasselman and Fulrath [36].

$$\frac{1}{E_0} = \frac{1}{E} \left(1 - \frac{3P(1-\sigma)(9+5\sigma)}{2(7-5\sigma)}\right) \quad (9)$$

$$\frac{1}{n_0} = \frac{1}{n} \left(1 - \frac{15p(1-\sigma)}{(7-5\sigma)}\right) \quad (10)$$

$$\sigma_0 = \frac{E_0}{2n_0} - 1 \quad (11)$$

$$K_0 = \frac{E_0 n_0}{3(3n_0 - E_0)} \quad (12)$$

The corrected values of E_0 , n_0 , K_0 and σ_0 values are given in Table-2. It can be seen from Table-2 that the values of E , n , E_0 , n_0 of the mixed Co-Zn ferrites are found to decrease continuously with the decrease of their cobalt content. The values of K_0 do not fluctuate systematically with composition and σ observed to be increasing with the decreasing of cobalt concentration. According to Wooster, the fluctuations of elastic moduli with concentration may be interpreted in terms of binding forces between the atoms of the ferrites. It may thus be deduced from the experimental values of E_0 and n_0 showed in Table-2 that the atomic binding between the ions of this cobalt-zinc ferrites decrease with decreasing cobalt concentration. So the inter-atomic binding between the various atoms is becoming continuously weekend. This compositional fluctuation of the elastic moduli is similar to what has been observed in

TABLE-2
ELASTIC DATA (UNCORRECTED) OF $\text{Co}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ AND ELASTIC DATA (CORRECTED) OF MIXED Co-Zn FERRITES

Ferrites	E_0 (GPa)	n_0 (GPa)	K_0 (GPa)	Poisson's ratio (σ_0)	V_1 (ms^{-1})	V_s (ms^{-1})	E (GPa)	N (GPa)	K (GPa)	Σ
CoFe_2O_4	47.19	18.79	32.13	0.25	9688	5582	41.85	16.73	28.09	0.25
$\text{Co}_{0.9}\text{Zn}_{0.1}\text{Fe}_2\text{O}_4$	43.88	17.8	27.34	0.23	9224	5482	39.49	16.1	24.12	0.22
$\text{Co}_{0.8}\text{Zn}_{0.2}\text{Fe}_2\text{O}_4$	34.99	14.14	22.19	0.23	8812	4896	31.77	12.84	24.48	0.23
$\text{Co}_{0.6}\text{Zn}_{0.4}\text{Fe}_2\text{O}_4$	27.19	10.47	22.48	0.29	8674	4264	25.07	9.69	27.18	0.29
$\text{Co}_{0.4}\text{Zn}_{0.6}\text{Fe}_2\text{O}_4$	18.21	6.68	22.15	0.36	8128	3418	16.83	6.21	26.85	0.35
$\text{Co}_{0.2}\text{Zn}_{0.8}\text{Fe}_2\text{O}_4$	15.70	5.70	21.3	0.37	7896	3226	15.04	5.51	25.7	0.36

various other ferrites [37,38]. The Debye temperature ϕ_D for the cobalt-zinc ferrites has been calculated using the simple method given by Anderson [39].

$$\phi_D = \frac{h}{k} \left(\frac{3N_A}{4\pi V_A} \right)^{1/3} V_m \quad (13)$$

where h is Planck's constant, k is Boltzmann's constant, N_A is Avogadro's number, V_A is the mean atomic volume and V_m is the mean sound velocity. Mean sound velocity is determined by

$$\frac{3}{V_m^3} = \frac{1}{V_1^3} + \frac{2}{V_s^3} \quad (14)$$

Here V_1 is the ultrasonic compressional velocity and V_s is the shear velocity. The reckoned values of ϕ_D and V_m are given in Table-3. A plot of ϕ_D against V_m is shown in Fig. 4, the values of ϕ_D varies linearly with V_m which has been observed in case noble metals [40,41].

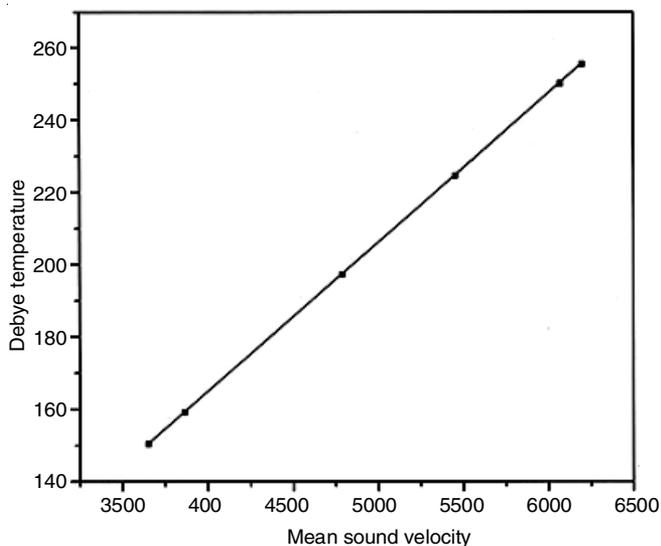


Fig. 4. Plot of Debye temperature against mean sound velocity

A linear dependence of V_1 on ρ for rocks having the same mean atomic weight was first proposed by Birch [42,43] and Simmons [44] has affirmed this evidence and has shown that V_s also varies linearly with density. Following this work anderson has evidenced that V_1/ρ and V_s/ρ are constant for a large number of oxide minerals having the same mean atomic weight. The values of V_1/ρ , V_s/ρ and the mean atomic weight (M/q) of the ferrites, calculated from the molecular weight and the number of atoms q in the chemical formula, the values are given in

Table-3. In the present probe, the values for the different ferrites are found to be from 33.52 to 34.24. It can be seen from Table-3 that the ratio's of V_1/ρ and V_s/ρ are nearly constant. This shows that the elastic behaviour of mixed Co-Zn ferrites is similar to that of oxide minerals. The variation of 'n' with 'E' is shown in Fig. 5. Here 'n' varies linearly with 'E' the same variation is observed with Ni-Zn ferrites.

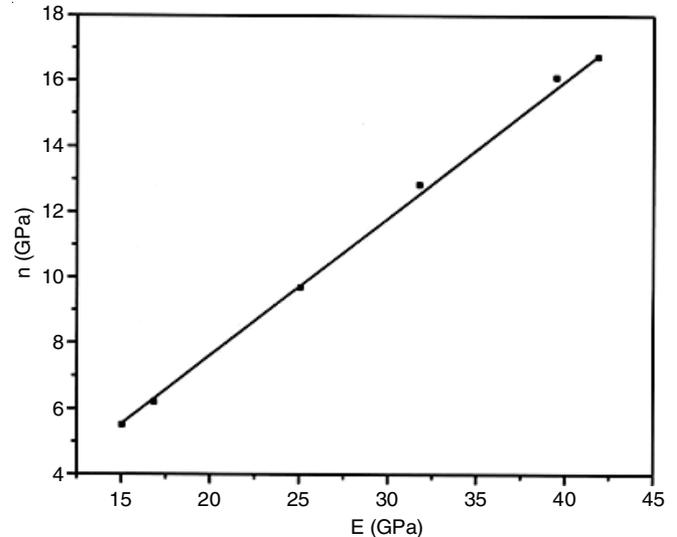


Fig. 5. Plot of rigidity modulus versus Young's modulus

Conclusion

It is concluded that the values of E , n , E_0 , n_0 of the mixed Co-Zn ferrites are found to decrease continuously with the decrease of their cobalt content. The values of K_0 do not fluctuate systematically with composition and σ observed to be increasing with the decreasing of cobalt concentration. According to Wooster, the fluctuations of elastic moduli with concentration may be interpreted in terms of binding forces between the atoms of the ferrites. It may thus be deduced from the experimental values of E_0 and n_0 that atomic binding between the ions of this cobalt-zinc ferrites decrease with decreasing cobalt concentration. So the inter-atomic binding between the various atoms is becoming continuously weekend. This compositional fluctuation of the elastic moduli is similar to what has been observed in various other ferrites.

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TABLE-3
DEBYE TEMPERATURE (ϕ_D), MEAN SOUND VELOCITY (V_m) AND
DEMONSTRATION OF V_1/ρ , V_s/ρ AND M/q FOR MIXED Co-Zn FERRITES

Ferrite	M/q	V_1/ρ	V_s/ρ	ϕ_D (K)	V_m (ms ⁻¹)
CoFe ₂ O ₄	35.52	1.804	1.039	258.67	6198.18
Co _{0.9} Zn _{0.1} Fe ₂ O ₄	33.61	1.720	1.022	253.32	6069.97
Co _{0.8} Zn _{0.2} Fe ₂ O ₄	33.70	1.644	0.913	227.57	5452.90
Co _{0.6} Zn _{0.4} Fe ₂ O ₄	33.88	1.627	0.800	199.82	4788.07
Co _{0.4} Zn _{0.6} Fe ₂ O ₄	34.06	1.527	0.642	161.31	3865.30
Co _{0.2} Zn _{0.8} Fe ₂ O ₄	34.24	1.489	0.608	152.40	3651.80

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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