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Synthesis and Structural studies of the Cu substituted Mg-Zn nano Crystalline ferro-spinels

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Abstract: *Nano crystalline Cu Substituted Mg-Zn ferrites having nominal compositional formula $Mg_{0.2}Cu_xZn_{0.8-x}Fe_2O_4$ ranging from x=0.0 to 0.8 with step increment of 0.2 were synthesized by citrate gel auto combustion method using nitrates of respective elements, citric acid as a fuel and by keeping 1:1 ratio of metal nitrates to citric acid. The synthesized powders of the investigated samples were sintered at 500^oC for 4 hours. X- ray diffraction patterns of all the investigated samples confirmed the single phase spinel structure which indicates the solubility of cations into their respective lattice sites. Structural parameters like crystallite size, lattice parameter, volume, hopping length for octahedral site and tetrahedral site are measured. The lattice parameter and crystallite size of the prepared samples were increases with the increasing the Cu concentration in the Mg-Zn ferrites. The surface morphology of the samples was observed by field emission scanning electron microscopy (FESEM).*

Keywords*: Nano ferrites, Citrate-gel method, XRD and FESEM.*

I. Introduction

The research on ferrites is very old but due to the their potential applications, even after so many decades researchers are still interested in the design of various types of ferrite materials substituted with different cations prepared various methods. Interest in nano crystalline ferrites has greatly enhanced in the past few years due to their importance in understanding the fundamentals of nano magnetism and their so many applications in high range storage device, ferro fluid technology, sensor technology, spintronics devices, target drug delivery, magneto caloric refrigeration etc [1]. The crystallite size reduces, finite size effects dominate the magnetic properties of the nano particles due to their surface to volume ratio. Based on the size and nature of interaction among the nano particles have a unique chance to exhibit different characteristic mechanisms like super-paramagnetism, super-ferro magnetism etc [2].

Nano crystalline particles with high surface to volume ratio exhibit conspicuous properties compared with their bulk particles. From the literature it is observed that ferrite nano particles exhibit high field magnetization irreversibility, small magnetization and modified magnetic moments with decrease in grain size. Along with the large surface to volume ratio, the redistribution of doped cations among the tetrahedral and octahedral sites from their normal site preferences could be the another reason for enhanced properties of the ferrite particles at the nano scale dimension.

Understanding and controlling the properties of nano crystalline ferrites is of interest not only for fundamental research such as understanding the quantum origins of magnetism, but also it is crucial for practical applications like high density information storage, ferrofluid technology, magnetocaloric refrigeration, magnetically guided drug delivery and magnetic resonance imaging (MRI) contrast enhancement agents. For example magnetic nano particles are super-paramagnetic at room temperatures in order to avoid agglomeration in biomedical applications.

From the survey it is observed among the several nano crystalline ferrite systems, Cu Substituted Mg-Zn ferrites with various copper concentrations has received sizable attention from past few years due to its high electrical resistivity, high magnetization, moderate permeability, chemical stability, and low magnetic & dielectric losses even at high frequencies [3-5]. Most of the researchers has prepared ferrites by the conventional ceramic process which is suffering from certain inherent problems like non uniform distribution, poor compositional control, chemical in-homogeneity etc. such non uniform particle distribution results in formation of voids or low density areas in the green body, hence on sintering one ends with the non reducible products [6].

II. Experimental techniques

Cu Substituted Mg-Zn ferrites having nominal compositional formula $Mg_{0.2}Cu_xZn_{0.8-x}Fe_2O_4$ ranging from x=0.0 to 0.8 with step increment of 0.2 were synthesized by citrate gel auto combustion method with following raw materials.

(1) Ferric nitrate
(ii) Magnesium nitrate
(iii) Cupric nitrate
(iv) Zinc nitrate
(v) Citric acid & Ammonia solution.

The detailed preparation method of citrate gel auto combustion method was explained in our earlier publication [7].The synthesized powders were sintered at 500^{o}C for 4 hours in air at a slow heating rate of 5^{o}C/min and then furnace cooled. X-ray diffraction analysis of the prepared ferrite powders were performed by using Philips diffractometer with CuK_{α} radiation with wavelength 1.5405A^{o}. The average crystalline size of the ferrites was determined from the measured width of their diffraction pattern using the Debye Scherer's formula [8]

$$D=0.91\lambda/\beta\cos\theta \quad (1)$$

Where λ is the wavelength of the X-ray used for diffraction,
β is the full width half maximum (FWHM) in radians.
θ is the diffraction angle.

The lattice constant was calculated using the following relation

$$2d\sin\theta = n\lambda \quad (2)$$

Where $d = \frac{a}{(h^2+k^2+l^2)^{1/2}}$ for fcc system.

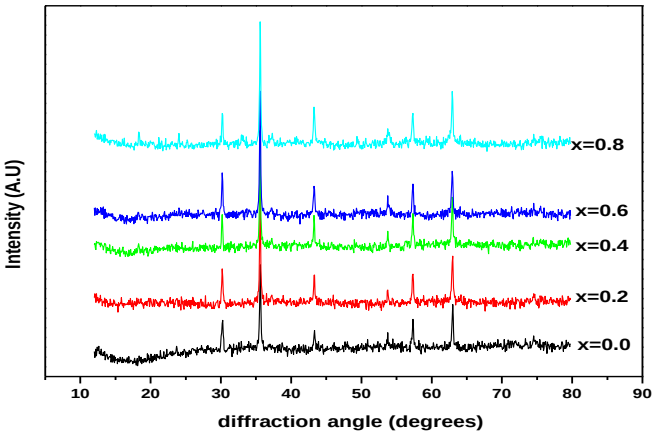
Hoping length for tetrahedral site $d_A=0.25a\sqrt{3}$ A^{o} (3)

And for octahedral site $d_B=0.25a\sqrt{2}$ A^{o} (4)

The surface morphology of the samples was observed by field emission scanning electron microscopy (FESEM).

III. Results and Discussions

The X-ray diffraction pattern of the prepared Cu Substituted Mg-Zn ferrites were shown in fig (1). The X-ray diffraction pattern of the prepared samples were confirmed the well defined homogeneous single phase cubic spinel structure without any impurity peak belonging to the space group Fd3m. Crystallite size of the prepared nano samples measured from the x-ray analysis was in the range 28-46nm.The strong diffraction from the (220),(311),(400),(422),(511) and (440) planes confirm the pure spinel phase of the annealed ferrites [9-10]. Lattice parameter of the prepared samples was increased with increasing the Cu composition Mg-Zn ferrites. Hoping length of A-site and B-site was observed to be increased with increasing with the Ni composition this is because hoping length of sites were proportional to the lattice parameters of the samples.



Fig(1). XRD pattern of Cu Substituted Mg-Zn ferrites

Table 1: Structural parameters of the prepared Cu Substituted Mg-Zn ferrites

Cu content	lattice Constant (A^{o})	crystallite size (nm)	Hoping length (d_A) in A^{o}	Hoping length (d_A) in A^{o}	Volum (A^{o})^3
x=0.0	8.356	28.92	3.618	2.953	583.48
x=0.2	8.356	31.54	3.618	2.954	583.60
x=0.4	8.358	35.35	3.619	2.954	584.02
x=0.6	8.361	41.90	3.620	2.955	584.65
x=0.8	8.368	43.30	3.623	2.958	586.10

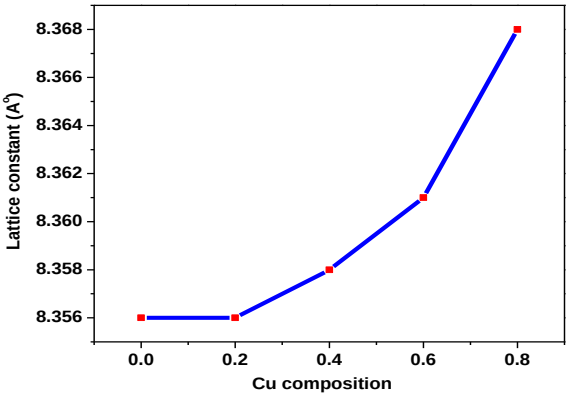


Fig (2) Variation of lattice constant with Cu composition

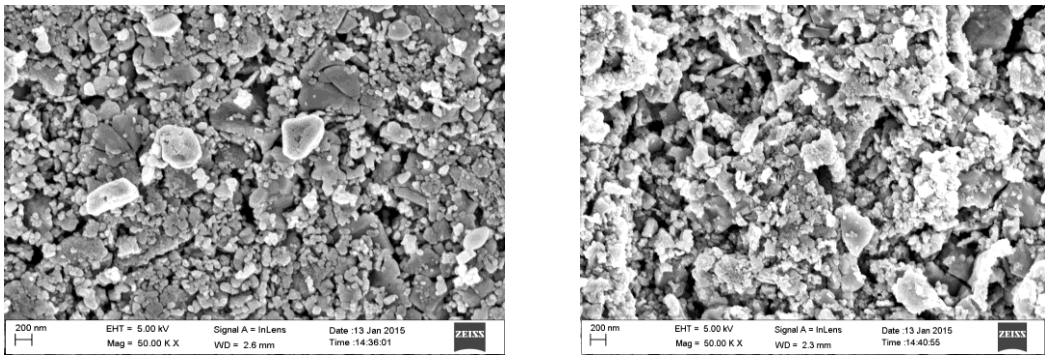


Fig (3) FESEM micrographs of Cu Substituted Mg-Zn ferrites

The surface morphology of the Cu Substituted Mg-Zn ferrites particles (x=0.0 and 0.8) sintered at 500^{o}C was examined by Field Emission Scanning Electron Microscopy (FESEM) shown in Fig (3) which indicates the agglomerated nano particles which is attributed to the interaction between the magnetic nano particles. Surface morphology of remaining samples also having same behavior (not shown).

IV. Conclusions

Citrate gel auto combustion method is the suitable technique for the preparation of nano crystalline Cu Substituted Mg-Zn ferrites at low temperature sintering. The X-ray diffraction pattern of the prepared samples were confirmed the well defined homogeneous single phase cubic spinel structure without any impurity peak belonging to the space group Fd3m. Lattice parameter, hopping lengths and volume of the unit cell of the samples increases with increasing the cu concentration in the Mg-Zn ferrites. FESEM micrograph shows the non-uniform distribution of particles with agglomerated particles.

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