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STRUCTURAL AND MAGNETIC PROPERTIES OF Cu SUBSTITUTED Ni-Mg-Zn FERRITE BY SOL-GEL METHOD

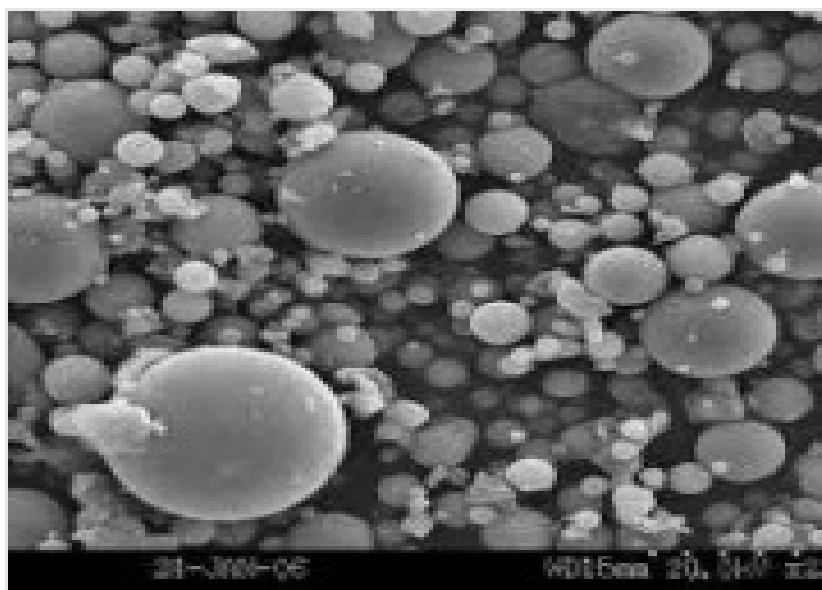


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Short Profile

S. R. Nevase is a Research Scholar in Jjt University, Jhunjhunu, Rajasthan.



ABSTRACT:

Ferrite nanoparticles of composition  $(\text{Ni}_{(0.5-x)}\text{Cu}_x\text{Mg}_{0.3}\text{Zn}_{0.2}\text{Fe}_2\text{O}_4)$  with  $x = 0.1, 0.2$  are synthesized by sol gel method and are characterized for structural properties using X-ray diffraction (XRD), Scanning electron microscopy (SEM), Transmission electron microscopy (TEM) and Fourier transform infrared spectroscopy (FT-IR). XRD analysis of the samples sintered at  $400^\circ\text{C}$  for 4h shows the cubic spinal structure for ferrites with size distribution from 30 to 50 nm. The samples exhibit ferro

magnetic behaviour, having saturation magnetization and coercivity values in the range 62.862- 71.811 emu/gm and 90.335-99.806 emu/gm respectively.

KEYWORDS

XRD, SEM, EDS and magnetic properties.

## INTRODUCTION :

The polycrystalline Ni Mg Cu Zn soft ferrites are suitable for core materials in micro inductor applications. The most significant and popular applications of ferrite in optics, electronics, mechanics and other technical fields. Nano-ferrites with copper substitution synthesized using sol-gel method are studied in the present work. The sol-gel technique of preparing nanoferrite has many significant advantages such as good stoichiometric control and for the production of ultrafine particle with nano size distribution in relatively short processing time at lower temperature.

The properties of nanoparticles varied from its morphology, size and microstructure and it is important to analyze obtaining conditions and method of synthesis of nanoparticles as well as synthesis of new metal oxides with new properties.

Specially we characterize structural properties of doped samples at room temperature and sintered at 400 °C. The various techniques are employed x-ray diffraction (XRD), field emission scanning electron microscope (FE-SEM), fourier transform Infra-Red (FTIR) spectroscopy, Transmission Electron Microscope (TEM), LCR-Q meter and vibrating sample magnetometer (VSM).

The aim of this research was to obtain Ni Cu Mg Zn ferrite nanoparticles. In this work we analyzed the morphological properties of  $(\text{Ni}_{(0.5-x)}\text{Cu}_x\text{Mg}_{0.3}\text{Zn}_{0.2})\text{Fe}_2\text{O}_4$  powders synthesized by sol-gel technique.

## EXPERIMENTAL:-

$(\text{Ni}_{(0.5-x)}\text{Cu}_x\text{Mg}_{0.3}\text{Zn}_{0.2})\text{Fe}_2\text{O}_4$   $x=0.1, 0.2$  nanoferrites were prepared by sol-gel method. In this method each sample was prepared by taking desired proportion of precursor nitrates. The precursor solution was prepared using AR grade metal nitrates  $\text{Ni}(\text{NO}_3)_2$ ,  $\text{Cu}(\text{NO}_3)_2$ ,  $\text{Mg}(\text{NO}_3)_2$ ,  $\text{Zn}(\text{NO}_3)_2$  and  $\text{Fe}(\text{NO}_3)_2$ . The nitrates were initially dissolved separately in minimum amount of water. The precursor solution was prepared by adding all above solutions and continuously stirred for 30 minutes at 80 °C. An aqueous solution of citric acid mixed with metal nitrate solution. A required amount of ammonia was slowly added into solution in order to adjust the pH value to about 7 since base catalysts are employed in order to speed up the reaction. The mixed solution was kept on to a hot plate with continuous stirring at 100 °C. After 3 to 4 hours all water molecules were removed from the mixture. The viscous gel has formed. The gel automatically ignited and burns with glowing flints. The auto combustion was completed within a minute. The result was the brown coloured ashes termed as precursor. The prepared powders were sintered at 400 °C for four hours to get the final powders for characterization.

## CHARACTERIZATION: -

Powder x-ray diffraction (XRD) pattern was carried out on a x-ray diffractometer (model Bruker D8) with Cu k irradiation ( $\lambda=1.5405\text{\AA}$ ). The lattice parameters, crystalline (grain) size of samples were calculated from XRD data. The scanning electron microscope (FEG-SEM) JSM-7600F and transmission electron microscope PHILIPS(model CM200) was used to study the morphology and estimate grain size. The infrared spectra of all samples were recorded in the range 1200-200cm in FTIR instrument(JASCO MODEL V 670). For magnetic studies M-H curves are obtained by using vibrating sample magnetometer.

STRUCTURAL CHARACTERIZATION :-

The structural characterization was carried out using diffractometer Bruker D8 with advanced system with a diffracted beam monochromatic Cu ka radiation( $\lambda=1.5405\text{\AA}$ ) source between the Bragg Angles  $20^\circ$  to  $80^\circ$  in steps of  $0.04^\circ/\text{sec}$ . The  $2\theta$  Vs intensity data obtained from this experiment and plotted graphs are as shown in fig.1 and fig.2. All Bragg reflections have been indexed which confirm the formation of cubic spinal structure in single phase. The strongest reflection comes from the (311) planes, which denote the spinal phase. The peaks indexed to (220) (311) (400) (422) (333) and (440) planes of cubic unit cell. All planes are allowed planes which indicate the formation of cubic spinal structure in single phase.

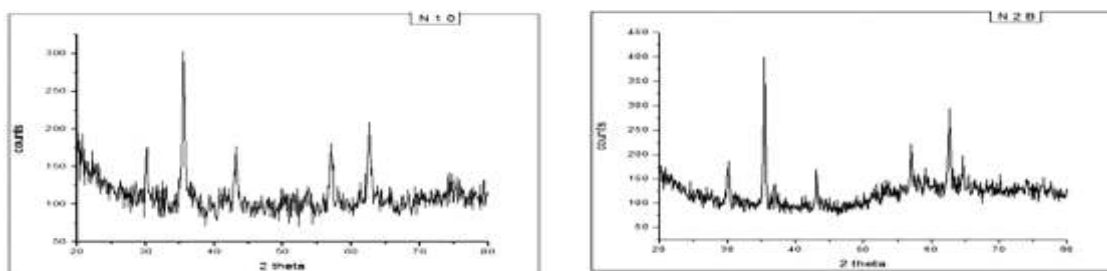


Fig.1 XRD pattern of the samples sintered at  $400^\circ\text{C}$  of  $(\text{Ni}_{(0.5-x)}\text{Cu}_x\text{Mg}_{0.3}\text{Zn}_{0.2})\text{Fe}_2\text{O}_4$  For  $x=0.1$

The particle size ( $d$ ) was calculated for all the compositions using the high intensity 311 peak and using

Scherer formula.

$$d = \frac{0.91 \lambda}{\beta \cos \theta} \quad \text{----- (1)}$$

Where,  $\lambda$  the wavelength of x-ray radiation,  $\beta$  is Full Width Half Maxima(FWHM) corresponding to maximum peak,  $\theta$  angle at peak position.

Table 1: composition ( $x$ ), Particle size ( $d$ ), Lattice constant ( $a$ ), Volume( $V$ ), X-ray density  $d_x$ , specific surface area ( $S$ ) and porosity ( $P$ ) of spinal ferrite system

$x$	$d$ nm	$a$ $\text{\AA}$	$V$ $(\text{\AA})^3$	$d_x$ $\text{gm/cm}^3$	$S$ $\text{m}^2/\text{gm}$	$P$
<b>0.1</b>	<b>36.83</b>	<b>8.3805</b>	<b>588.595</b>	<b>5.098</b>	<b>32.95</b>	<b>61.9079</b>
<b>0.2</b>	<b>49.41</b>	<b>8.3825</b>	<b>589.018</b>	<b>5.078</b>	<b>36.14</b>	<b>63.2167</b>

parameter( $a$ ) for all the composition of Ni Cu Mg Zn nanoferrites have been calculated from the values of  $d$ -spacing's and are given in table 1.

The lattice parameter ( $a$ ) of individual composition was calculated by using a formula.

$$a = d_{hkl} \sqrt{h^2 + k^2 + l^2} \quad \text{----- (2)}$$

Where, a= Lattice constant, (hkl) are the miller indices of crystallographic plane ,  
 d= inter planner spacing

SEM Analysis: -

Performing scanning electron microscope (SEM) with model-JSM-7600F microscope. We analyzed the structure of Ni Cu Mg Zn Fe<sub>2</sub>O<sub>4</sub> powder and show typical micrographs in fig.3

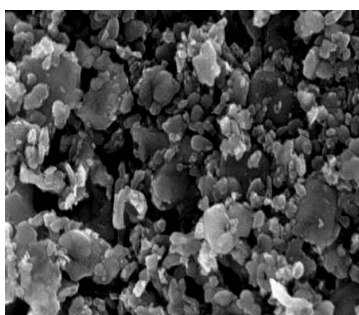


Fig.(a) x = 0.1

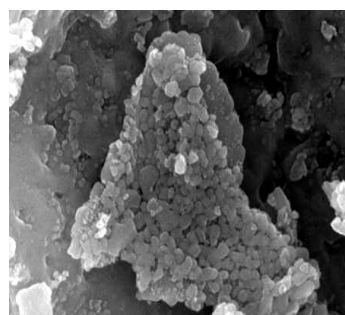


Fig. (b) x = 0.2

Fig.3: SEM micrographs

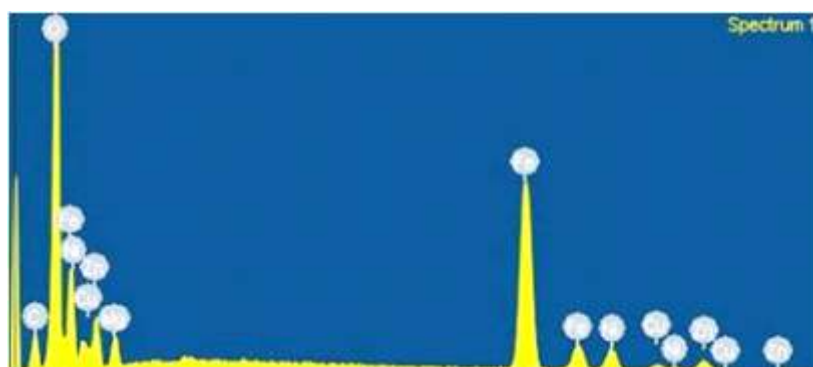


Fig 4: EDS image

It can be seen from SEM micrographs of various compositions that the morphology of particles is very similar. They indicate that particle size of sample lies in the nanometer range having a spherical shape and narrow size

The energy dispersive scattering (EDS) pattern obtained for all the samples .It may be seen that besides the characteristics peaks of Fe,Ni,Cu,Mg,Zn, peaks arising out of the substrate are Carbon and Oxygen.

## Magnetic studies

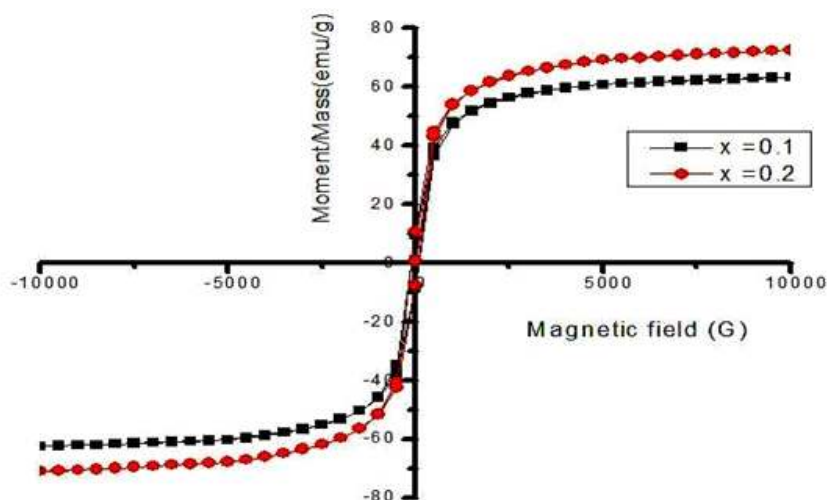


Fig 5: Magnetic hysteresis loops drawn between magnetic field and magnetic moment

Table 2 : Magnetic properties

sample	Ms (emu/gm)	Hc (G)	Mr (emu/gm)
X=0.1	62.862	99.806	8.8733
X=0.2	71.811	90.335	9.2039

To find saturation magnetization, coercivity and remanance, M-H curves are obtained with vibrating sample magnetometer. The values of saturation magnetization (Ms), coercivity (Hc) and remanance (Mr) for  $\text{Ni}_{(0.5-x)}\text{Cu}_x\text{Mg}_{0.3}\text{Zn}_{0.2}\text{Fe}_2\text{O}_4$  have been determined and are listed in Table 2

The figure 5 shows tall, narrow hysteresis loops with small loop areas. Therefore the mixed ferrites of different combinations are referred as soft ferrites.

## CONCLUSION:-

The sol-gel method is convenient for the synthesis of nanosize Ni Cu Mg Zn ferrite. X ray diffraction pattern confirm that the synthesis of fully crystalline Ni-Mg-Cu-Zn ferrite nanoparticles at high temperatures. The particle sizes of the nanoparticle samples were found to be about 30 to 50 nm on sintering at 400°C. The dielectric constant ( $\epsilon'$ ), dielectric loss ( $\epsilon''$ ) and dielectric loss tangent (tan $\delta$ ) decreases as frequency increases.

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