ISSN : 2393-8188 (print)
2393-8196 (online)
www.milliyasrcollege.org.journal.php

Dielectric and Structural study of Ni-Cu-Mg-Zn Nanoferrite

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Abstract : Ferrite materials with chemical formula $Ni_{0.3}Cu_{0.2}Mg_{0.3}Zn_{0.2}Fe_2O_4$ have been synthesized by Autocombustion technique. The synthesis sample were characterized to investigate the structural, morphological and dielectric properties. The samples were sintered at 400 ^{0}C for 4 hr. The XRD measurements confirmed the formation of a single phase cubic spinel structure of ferrites with size distribution between 36 and 28 nm. The morphology studied using SEM technique. The dielectric constant, dielectric loss, and loss tangent at room temperature (28 ^{0}C) were measured using LCR-Q meter. By increasing frequency, the dielectric constant and loss tangent were decreases. Reaches to constant value shows the normal ferromagnetic behavior of ferrites materials.

Keywords : Structural properties, Dielectric properties, Morphological properties, Ferrite materials, etc**.**

1. INTRODUCTION :

Spinel ferrites with the general formula MFe_2O_4 (M is divalent metal cation) are attracted much attention in recent years because of their potential applications in high density magnetic recording, magnetic fluids, data storage, and gas sensors. The polycrystalline Ni, Mg, Cu and Zn soft ferrites are suitable for core materials in micro inductor applications. Ferrites have popular applications in the fields of optics, electronics, mechanics and other technical fields [1–3]. The autocombustion method for synthesis nanoferrite having number of merits for the production of ultrafine particle with nano-scale distribution in relatively short processing time at lower temperature and good stoichiometric control [4]. The properties of nanoparticles varied from its morphology, size and microstructure. In this present work, we analyzed and studied the structural, dielectric and morphological behaviour of $Ni_{0.3}Cu_{0.2}Mg_{0.3}Zn_{0.2}Fe_2O_4$ sample at (28 ^{0}C) room temperature by sintering at 400 ^{0}C by sol-gel method. The various characterization techniques such as XRD, FE-SEM, FTIR, and TEM were used to study the structural, dielectric and morphological behaviour of prepared samples.

2. MATERIALS AND METHOD :

Nonoferrite material $Ni_{0.3}Cu_{0.2}Mg_{0.3}Zn_{0.2}Fe_2O_4$ were synthesized by sol-gel autocombustion techniques with taking desired proportion of precursor nitrates. The precursor solution were prepared using pure AR-grade metal nitrates such as $Ni(NO_3)_2$, $Cu(NO_3)_2$, $Mg(NO_3)_2$, $Zn(NO_3)_2$ and $Fe(NO_3)_2$. Initially, these nitrates were dissolved individually in less amount of double filtered distilled

water and mixed all above solutions by continuously stirred at 80 ^{0}C for ½ hr. Citric acid solution added in metal nitrate solution and ammonia was slowly added into solution to control the pH as neutral. All water molecules were removed from the mixture by keeping solution on to a hot plate with continuous stirring at 80 ^{0}C about four hours. Automatically, the viscous gel burned and autocombustion process was completed. The result was the brown colored (ash) precursor by sintering at 400 ^{0}C for four hours for future study.

3. RESULTS AND DISCUSSION :

3.1 Structural Analysis : X-Ray diffraction patterns were taken at room temperature to confirm the nanocrystal structure of the synthesized samples. The XRD patterns were recorded in the 2θ range from 20^0 to 80^0 using Cu-kα radiation (λ = 1.5406 Å) with scanning rate 2^0 per/m. The lattice parameters, crystalline (grain) size of samples were calculated from XRD data. The 2θ Vs intensity data obtained from this experiment and plotted graph as shown in figure-1.

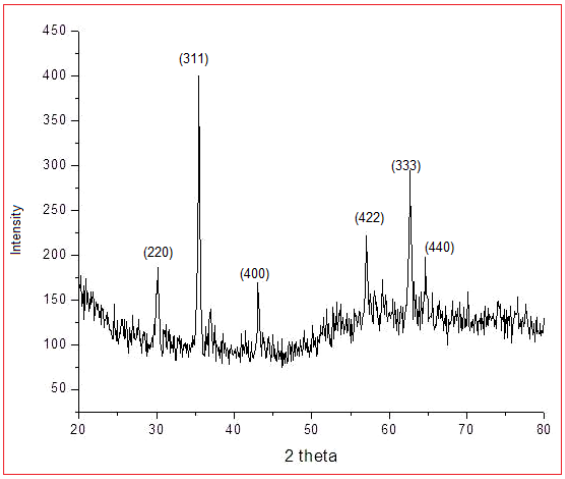

Figure-1: X-ray pattern of $Ni_{0.3}Cu_{0.2}Mg_{0.3}Zn_{0.2}Fe_2O_4$

Indexed Bragg reflections has confirming the formation of cubic spinal structure in single-phase with highest reflection from (311) hkl plane. The peaks such as (220), (311), (400), (422), (333), and (440) planes are indexed to cubic unit cell and indicates the formation of cubic spinal structure in single-phase. The particle size (d) was derived for the composition using Scherer formula, such as

$$d = \frac{0.91\,\lambda}{\beta cos\theta}$$

Where, λ is the wavelength of X-ray radiation, β is FWHM (full width half maxima) related to highest peak and θ is the angle at peak position.

Table: Composition(x), Particle Size(d), Lattice Constant(a), Volume(V), X-ray Density(d_x), and Specific Surface Area(S) for prepared spinal ferrite system

x	d nm	a A^0	V $(A^0)^3$	d_x gm/cm^3	S m^2/gm
0.1	35.62	8.3724	586.8773	5.112	32.95

Synthesis parameters for the composition of $Ni_{0.3}Cu_{0.2}Mg_{0.3}Zn_{0.2}Fe_2O_4$ nanoferrites were calculated using the values of d-spacing's and noted in above table. The lattice parameter (a) of individual composition was designed by using a formula.

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

Where, 'a' is Lattice constant, (hk*l*) are the miller indices of crystallographic plane, and d=inter planner spacing.

3.2 Morphological Analysis :

The synthesized sample was characterized by FE-SEM with model-JSM-7600F microscope to study morphology of sintered sample. The analyzed the structure of $Ni_{0.3}Cu_{0.2}Mg_{0.3}Zn_{0.2}Fe_2O_4$ nanoferrite powder shows typical micrographs in figure-2.

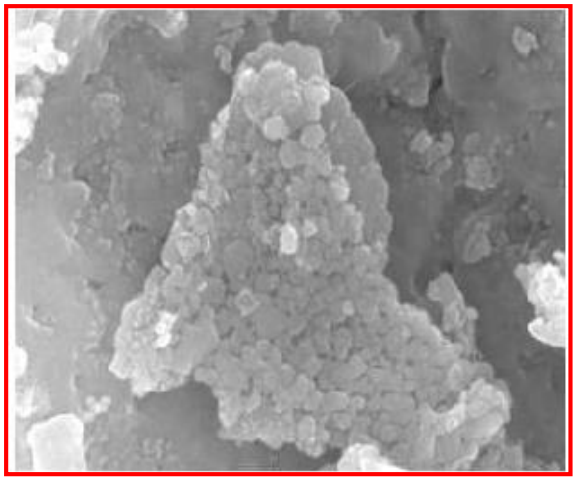

Figure-2: Morphology of $Ni_{0.3}Cu_{0.2}Mg_{0.3}Zn_{0.2}Fe_2O_4$ ferrite

From the FE-scanning electron microscopy, micrograph of composition indicate that particle size of sample lies in the nanoscale range having a spherical shape and narrow size distribution.

The energy dispersive scattering (EDAX) pattern shows that besides the peaks of Iron (Fe), Nickel (Ni), Copper (Cu), Magnesium (Mg), and Zinc (Zn) also Carbon and Oxygen peaks arises for the prepared powder.

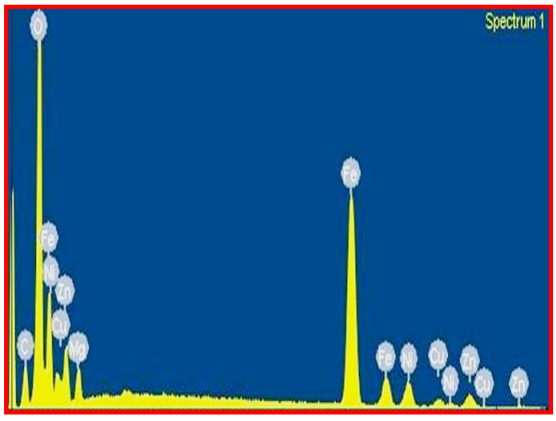

Figure-3: EDAX pattern of $Ni_{0.3}Cu_{0.2}Mg_{0.3}Zn_{0.2}Fe_2O_4$

3.3 Dielectric Analysis :

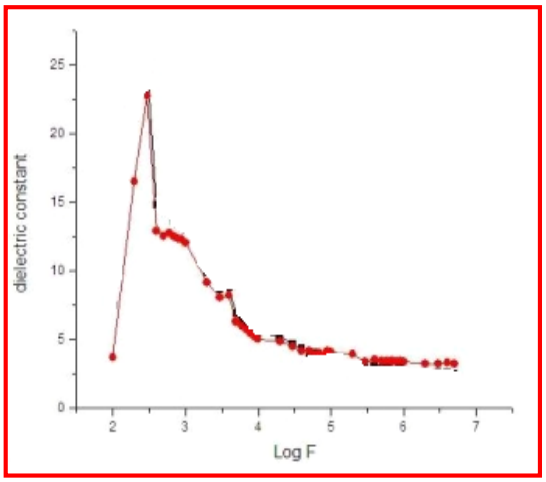

Figure-4(a): Change in dielectric constant with respect to *log(f)* of $Ni_{0.3}Cu_{0.2}Mg_{0.3}Zn_{0.2}Fe_2O_4$

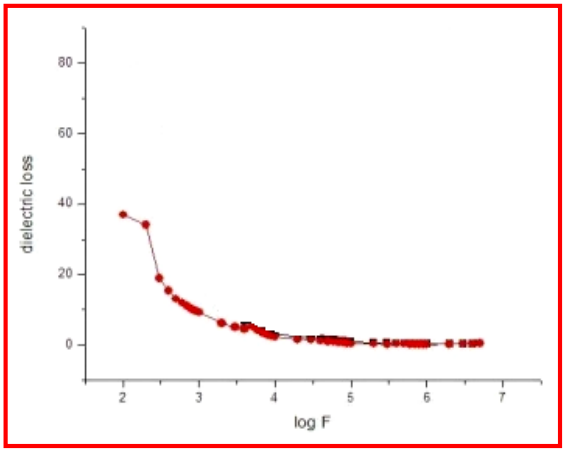

Figure-4(b): Change in dielectric loss with respect to *log(f)* of $Ni_{0.3}Cu_{0.2}Mg_{0.3}Zn_{0.2}Fe_2O_4$

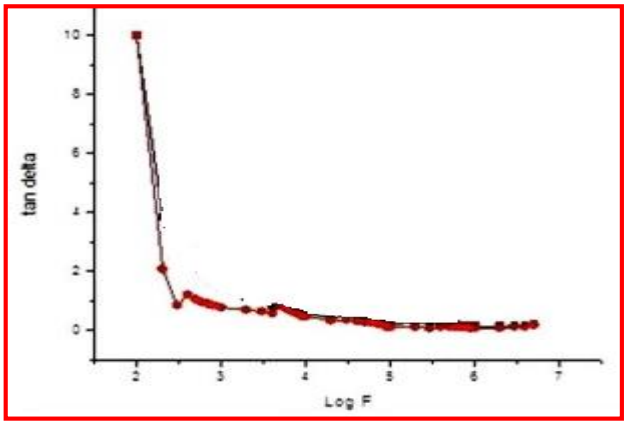

Figure-4(c): Change in dielectric loss tangent with respect to *log(f)* of $Ni_{0.3}Cu_{0.2}Mg_{0.3}Zn_{0.2}Fe_2O_4$

In the frequency range between 100 Hz to 5MHz, the dielectric constant (ε'), dielectric loss (ε'') and dielectric loss tangent (tan δ) were measured by using

LCR-Q meter. The measurements of these parameters were carried out at 28 ^{0}C room temperature on a disc shaped pellets using 2-probe method. Figure-4(a) indicates that dielectric constant (ε') decreases with increase in *log*(*f*) frequency and the behavior of frequency dependence of dielectric constant (ε') is noble covenant with the well-known spinal ferrites. It is also observed that the decrease in polarization is with increase in frequency. Figure-4(b) indicates that dielectric loss (ε'') decreases with increase in *log*(*f*) frequency. Figure-4(c) indicates that dielectric loss tangent shows a regular dielectric behaviour with frequency *log*(*f*). By increases frequency beyond certain frequency, exponentially tangent loss (tanδ) decreases and the hopping frequency of the charge carrier does not changes the polarity of the external field. Hence, there is a tough co-relation between the dielectric behaviour and the conduction mechanism of ferrites.

4. CONCLUSION :

The autocombustion technique is a convenient for the synthesis of $Ni_{0.3}Cu_{0.2}Mg_{0.3}Zn_{0.2}Fe_2O_4$ nanoferrite. From structural analysis, XRD-pattern of prepared nanoparticle sample confirm that the synthesis of fully crystalline Ni-Mg-Cu-Zn ferrite nanoparticles at high temperatures. From morphological analysis, nanoparticle size of the sample were observed between 36 to 28 nm on sintering at 400 °C. From dielectric analysis, dielectric constant (ε'), dielectric loss (ε'') and dielectric loss tangent (tan δ) decreases as frequency increases. The constant value of dielectric properties shows the normal ferromagnetic behavior of ferrites materials.

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