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SINTERING EFFECT ON STRUCTURE AND MORPHOLOGY OF $MG_{0.2}CU_{0.5}ZN_{0.3}$ FERRITE USING SOL-GEL METHOD

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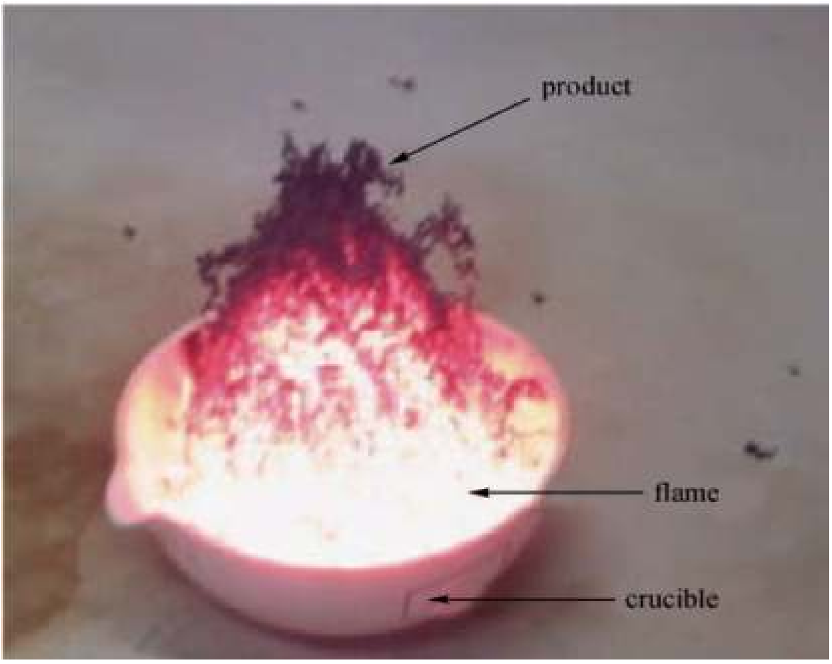
Short Profile

M. T. Sonawane is Research Scholar at Jagadishprasad Jhabarmal Tibrewala University of Jhunjhunu , Rajasthan , India. He has completed M.Sc. and D.H.E. He has Professional Exprience 30 years. He has done one project on Study on nanocrystalline MgCuZn ferrite materials.



ABSTRACT:

Ferrite sample of $Mg_{0.2}Cu_{0.5}Zn_{0.3}$ Fe_2O_4 nanocrystalline powder was synthesized by Sol-gel auto-combustion method. The synthesized powder was sintered at two different temperatures 4000C and 7000C for four hours to obtain two samples. Structural, compositional and phase properties of samples were studied by X-ray diffraction (XRD) technique. The X-ray diffraction study confirmed the formation of single phase cubic spinel structure of samples. The average crystallite sizes of these ferrite powders were determined from XRD patterns by using Scherrer formula which confirmed the nanosize of particles. Lattice constant was determined by the standard formula. The Fourier Transform Infrared Spectroscopy (FTIR) confirmed the formation of ferrite. The morphological investigations and sizes of the samples were studied by using scanning electron microscopy (SEM). .

KEYWORDS

Sol-gel auto combustion , Nanocrystalline MgCuZn ferrite , FTIR , SEM, XRD.

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1.INTRODUCTION :

The latest electronic devices such as cellular phones, video cameras, notebook computers, hard and floppy drives etc require chip inductors as important passive surface mount devices (SMD) because of their small dimensions, lightweight and better functions [1, 2]. The traditional wire-wound chip inductors can only be miniaturized to a certain limit and lack of magnetic shielding leads to the synthesis of new materials for the multilayer chip inductors. Only NiCuZn ferrites were developed as the material used in the chip components [3, 4]. The NiCuZn ferrites are comparatively sensitive to stress and their magnetic properties get easily changed by the stress caused at the internal electrode. Silver is generally used as the material for the internal conductor of the multilayer chip inductors (MLCI) due to its low resistivity, resulting in the components with high quality factor Q [3]. Also, Ag paste is commercially available at lower cost. Since the melting point of silver is 961°C, the sintering temperature of ferrite used for the manufacture of chip inductor should be below 940°C. This prevents Ag diffusion into the ferrite that would increase the resistivity of the internal conductor. Moreover, the segregation of Cu^{+2} from the ferrite induced by the diffused Ag can be avoided and magnetic properties of the material remain intact.

MgCuZn ferrites were found to be more suitable [5, 6]. Normally, MgCuZn ferrites were sintered at temperatures higher than 1000°C [5-7]. In order to use these ferrites in multilayer chip components, the sintering temperature must not be over the melting point of Ag. Low temperature sintering of MgCuZn ferrite is required for MLCI applications.

Wet chemical methods such as hydrothermal synthesis [8] , combustion synthesis [9,10] , sol–gel technique [11] , citrate method [12,13] and chemical co-precipitation method [14] have been developed for preparation of nanosized ferrites. However, all of these wet chemical methods, to some extent, still need calcination at relatively high temperatures and long soaking to obtain the final powders with expected crystal structure. The sol–gel auto combustion method was developed to synthesize ferrite nanocrystalline powders. This is a way with a unique combination of the chemical sol–gel process and the combustion process. The process has advantages of inexpensive precursors, simple preparation and resulting nanocrystalline homogeneous powder. In present study the sol–gel auto combustion preparation of MgCuZn ferrite nanocrystalline powder with composition $Mg_{0.2}Cu_{0.5}Zn_{0.3}Fe_2O_4$ was carried out using aqueous solution of ferric nitrate, magnesium nitrate, zinc nitrate, copper nitrate, citric acid and ammonia. The phase evolution during combustion and the subsequent calcination was also investigated. This method resulted in formation of single phase ferrite nano crystalline powder after combustion .These samples were characterized by FTIR spectra, XRD, EDS and SEM techniques.

2. EXPERIMENTAL PROCEDURE

2.1 Synthesis of Samples

The magnesium nitrate [Mg $(NO_3)_2$ H_2O], zinc nitrate [$Zn(NO_3)_2$ $6H_2O$], copper nitrate [$Cu(NO_3)_2$ $3H_2O$] , iron nitrate [$Fe(NO_3)_3$ $9H_2O$] and citric acid [$C_6H_8O_7$ H_2O] of analytical grade were used to prepare the ferrite composition $Mg_{0.2}Cu_{0.5}Zn_{0.3}Fe_2O_4$. Nitrates were initially dissolved separately in distilled water and stirred well for 20 minutes at 800 C. The precursor solution was prepared by adding all above solutions and continuously stirred for 30 minutes at 80° C. The molar ratio of nitrates to citric acid was 1:1. Then ammonia solution was slowly added to adjust the pH at 7. The mixed solution was kept on to a hot plate with continuous stirring at 100° C. When finally all water molecules were removed from the mixture, the viscous gel began to transform in to fluppy loose powder. The auto combustion was completed within a minute. The prepared powder was sintered at 400°C and 700° C for four hours to get the final samples.

2.2 Characterization

Fourier Transform Infrared spectra (FTIR) of calcined powders at 400°C and 700°C were recorded on a Bruker (Tensor 27) spectrophotometer from 400 to 4000 cm^{-1} .The phase identification of the dried gel, as-burnt powder and calcined powders was performed by X-ray diffraction (XRD) on a Philips PW-1730 X-ray diffractometer using CuKα radiation (? = 1.5405 A°). The average crystallite size of the synthesized powders was determined by X-ray line

broadening technique using the well known Scherrer formula. The elemental composition of the powders was analyzed by EDS analysis. Scanning electron microscopy (SEM) was used to determine the microstructure of the sintered specimens.

3. RESULTS AND DISCUSSION

3.1 XRD Analysis

Fig. 1 shows XRD patterns of two samples of $Mg_{0.2}Cu_{0.5}Zn_{0.3}Fe_2O_4$ ferrite prepared by sol-gel auto-combustion method sintered at 400°C and 700° C. The diffraction peaks give the evidence of the formation of crystalline spinel ferrite phase in the samples. The peak position and relative intensity of all diffraction peaks match well with the previous results of researchers. The peaks correspond to the planes (220), (311), (440), (422), (333) and (440).

The average crystallite size was calculated from full width at half maximum (FWHM) for main 311 peak using Scherrer formula:

$$t= \frac{0.9\lambda}{\beta cos\theta}$$

where , t = grain size in nm , ß = full width at half maxima, ? = Bragg's angle for (311) peak. The crystalline size (t) of powders is in the nano range. The observations show that the particle size increases with increase in sintering temperature.

The d-spacing for recorded peaks were calculated using Bragg's law. The value of the lattice constant 'a' for the samples was determined from the position of principal (311) peak using

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

where, h, k, l are the Miller indices of (311) plane. The observed values of lattice constant (a) are listed in Table(1)

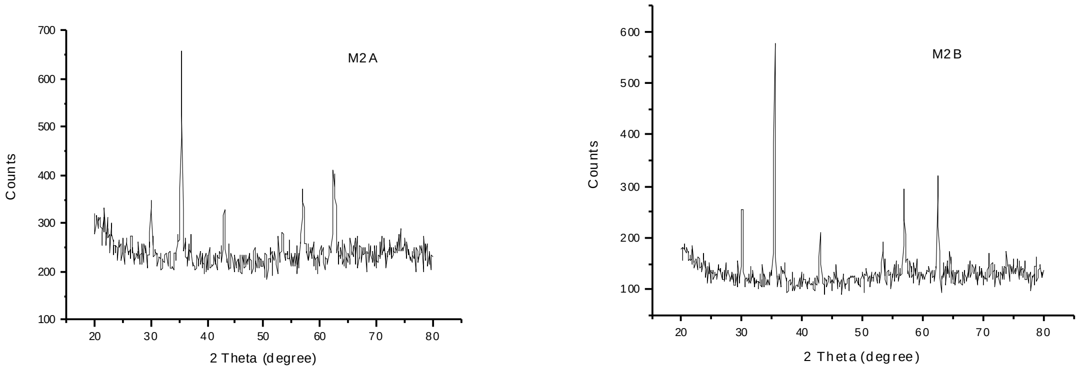

Fig.1 :XRD patterns of $Mg_{0.2}Cu_{0.5}Zn_{0.3}Fe_2O_4$ ferrite sintered at 400^0C and 700^0C

Table 1: Particle size (t) , Lattice constant (a) and Volume(V) of spinel ferrite $Mg_{0.2}Cu_{0.5}Zn_{0.3}Fe_2O_4$

Sintering Temperature	**Particle size t(nm)**	**Lattice Constant a($A^{0)}$**	**Volume $(A^0)^3$**
400^0C	41.18	8.3949	591.63
700^0C	56.08	8.4078	594.36

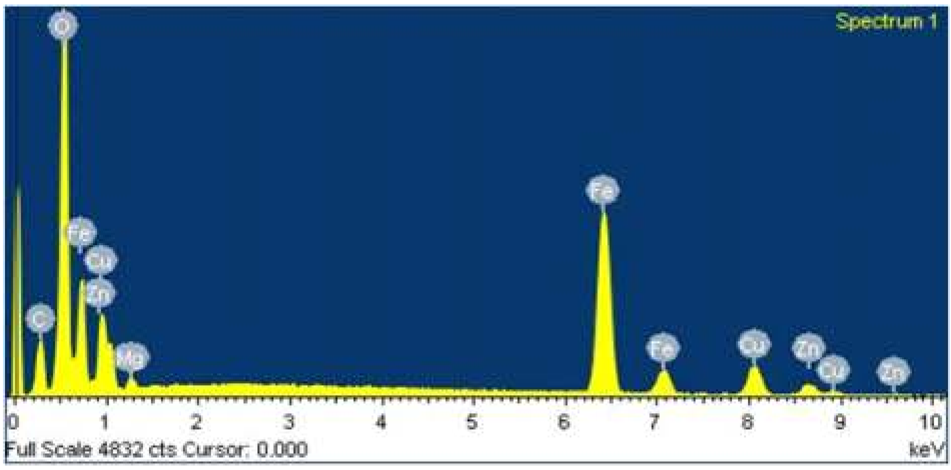

Fig.2 : EDS Pattern of $Mg_{0.2}Cu_{0.5}Zn_{0.3}Fe_2O_4$ ferrite

The elemental composition of the powder was analyzed by EDS analysis. The EDS spectra (fig.2) indicates the presence of mainly Mg, Cu, Zn, Fe and O with small amount of carbon.

3.2 FTIR Spectra :

Fig.3 represents fourier transform infrared spectra of the ferrite. $Mg_{0.2}Cu_{0.5}Zn_{0.3}Fe_2O_4$.

The FTIR spectra of the powder thermally treated at 700^0C was recorded (Fig.3) in order to confirm the formation of the spinel phase and to understand the nature of the residual carbon in the sample. The IR absorption bands of solids are usually assigned to the vibrations of ions in the crystal lattice. Two main metal-oxygen bands are seen in the IR spectra of all spinel ferrites. The sample shows characteristic absorptions of the ferrite phase with a strong absorption around 680 cm^{-1} and weak absorption in the range 420 - 450 cm^{-1}. This difference in the band positions expected because of the difference in the metal-oxygen (M^{n+}-- O^{2-}) distance for the octahedral and tetrahedral sites.

Waldron [15] reported the vibrational spectra of ferrites and attributed the sharp higher absorption band around 600 cm^{-1} to the intrinsic vibrations of the tetrahedral sites and the other lower absorption band around 447cm^{-1} to that of the octahedral sites. In Fig. 3, two weak and broad absorptions around 1400 and 1600 cm^{-1} correspond to the presence of small amounts of residual carbon in the samples. These absorptions in the present powder are very weak which indicates that the residual carbon had mostly burnt away during the self-combustion process.

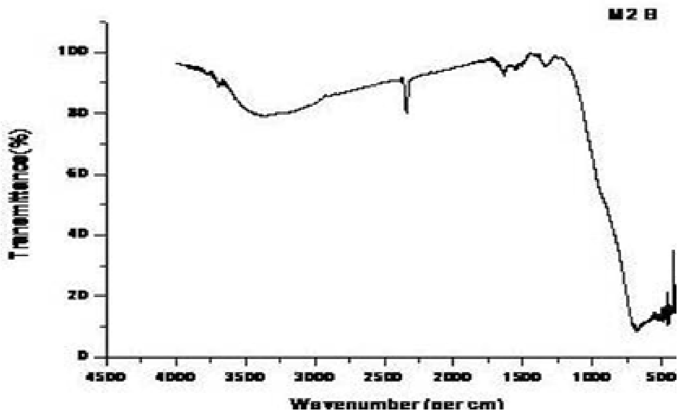

Fig. 3: FTIR Spectra of sample Mg0.2Cu0.5Zn0.3Fe2O4

3.3 Scanning Electron Microscopy (SEM):

Figure 4 shows the SEM microphotographs of two samples of $Mg_{0.2}Cu_{0.5}Zn_{0.3}Fe_2O_4$ ferrites sintered at 400^0C and 700^0C for 4 hours. The SEM images show the average particle sizes in nano range which are in agreement with XRD calculations. The average particle size increases with increase in sintering temperature.

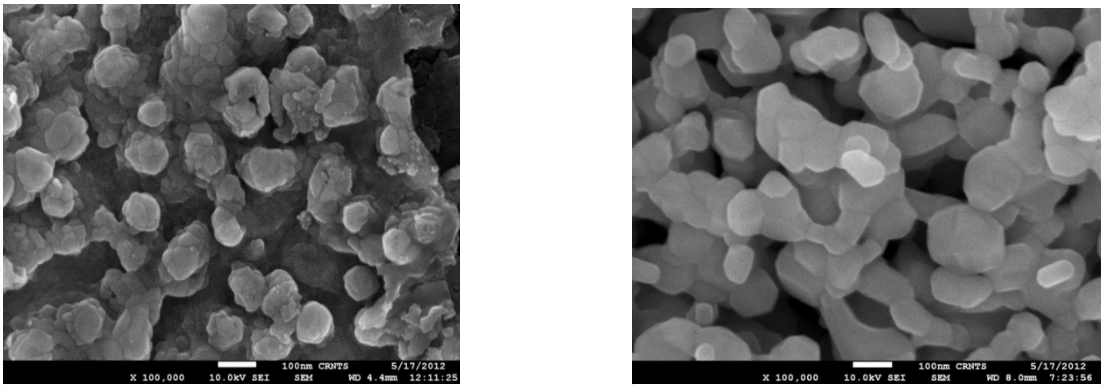

Fig.4 : SEM micrographs of $Mg_{0.2}Cu_{0.5}Zn_{0.3}Fe_2O_4$ ferrite sintered at 400^0C & 700^0C

4 . CONCLUSIONS

Nanocrystalline $Mg_{0.2}Cu_{0.5}Zn_{0.3}Fe_2O_4$ ferrite was successfully synthesized by sol-gel auto-combustion technique. XRD patterns of samples show the nanocrystalline spinel nature. The average particle size of samples increase with increase in sintering temperature which is supported by SEM micrographs. IR spectra confirmed the formation of single phase ferrite.

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