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Sol–gel auto-combustion synthesis, structural, IR and XRD properties of Nanocrystalline Mg Ni Zn Fe_2O_4 Spinal Ferrite

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Subject: Nanotechnology

Abstract

Nanocrystalline $Mg_{0.7-x}$ $Ni_xZ_{0.3}$ were synthesized through nitrate-citrate by sol-gel auto-Combustion method at relatively low temperature. It is unique combustion and the chemical gelation process. For this x =0.2, x =0.4 and x =0.6. The prepared powders were sintered at 700^0c for two hours. They are obtained as dried gel after the successful chemical reaction of their respective metal nitrate solution in the midst of citric acid as catalyst. The samples were characterized by X–Ray Diffraction (XRD), Scanning electron microscope (SEM), Infrared spectroscopy. (IR), The IR spectra confirmed that the synthesized material is ferrite. The X-Ray revealed that the size of the crystalline particle which was calculated by scherrer method is between 89.21nm to 92.87 nm.

Keywords*: Sol-gel, auto combustion, Nanocrystalline, Mg Ni Zn Fe_2O_4, IR, XRD, SEM*

Introduction

Nanotechnology is the study and design of systems at the nanometer scale [0.000000001(10^{-9}) meter] the scale of atoms and molecules. Human ability to manipulate materials on the nanoscale could revolutionize the way that almost everything is designed and made as nature does it. [1] Nonmaterial and nanotechnologies attract tremendous attention in recent researches. New physical properties and new technologies both in sample preparation and device fabrication evoke on account of the development of nanoscience. Various research fields including physics, chemists, material scientists and engineers of mechanical and electrical are involved in this research [2]. The use of material Mg Ni Zn Fe_2O_4 is in different areas. Up to this stage the research work on Mg Ni Zn Fe_2O_4 is very limited. The sol-gel route was used for synthesis of nanoferrite material. The research work about the synthesis of nanoferrites of Mg Ni Zn Fe_2O_4 is limited so to study XRD, SEM, IR topics. The use of Mg Ni Zn Fe_2O_4 is currently noticed as conductors in various applications [3]. The applications of nanomaterials can be historically traced back to even before the generation of modern science and technology. Nanoparticles were used as dye materials in ceramics by the peoples [4]. Colloidal gold was used in medical treatment for curve of dipsomania, arthritis etc. as early as from 19 countries. Systematic experiments conducted on nanomaterial had also been started from known Faraday experiments [5] in 1857. This so-gel route is sophisticated and easy to do, so I have select above cited method for synthesis of nanoferrite materials. The application of sol-gel processing is widespread and certain applications, such as coating on window glass has been used on commercial basis. Sol-gel technique has been implemented to prepare silver nanoparticles and silica nanospheres [6]. This method involves the hydrolysis. Many techniques have been provided for the synthesis of the nano-sized ferrites. These methods include sol–gel [7], The powders for x= 0.2, 0.4 & 0 .6 were synthesized by sol- gel auto combustion method. The chemicals used for the powders were magnesium nitrate [Mg $(No_3)_26H_2o$], Nickel nitrate [$Ni(No_3)26H_2o$], Zinc nitrate[$Zn(No_3)26H_2o$], Ferric nitrate[$Fe_2(No_3)9H_2o$] citric acid[$C_6H_8O_7H_2O$]. These chemicals were analytical grade. All were dissolved in procedure was carried out in air atmosphere without protection of inert gases. The metal nitrates were dissolved together in minimum amount of distilled water so as to get clear solution. An aquas solution of citric acid mixed with metal nitrate solution. Then Ammonia solution was added to adjust the PH 7. Then the solution was kept on to magnetic stirrer with continuous stirring by magnetic needle at 100^0c. During evaporation the solution became viscous and finally all water molecules were removed from the mixture. Then the viscous gel began frothing. After few minuets the gel automatically ignited and burns with glowing flints. The decomposition reaction would not stop before the whole citrate complex was consumed. The auto combustion was completed within a minute yielding the brown

colored ashes termed as precursor. The as prepared powders of all the samples were sintered at 700^0c for 2 hours to get the final product. The samples were in powder configuration for x-ray investigation. Part of the powder was x-ray examined by Philips x-ray diffract meter (model 3710) using CuK radiation (=1.5406). Powder x-ray diffraction studies (x-ray) have been carried out on the sintered samples at 400^0c and 700^0c for $Mg_{0.5}$ $Ni_{0.2}$ $Zn_{0.3}$ Fe_2O_4, $Mg_{0.3}$ $Ni_{0.4}$ $Zn_{0.3}$ Fe_2O_4, $Mg_{0.1}$ $Ni_{0.6}$ $Zn_{0.3}Fe_2O_4$. The dried gel powder is amorphous. The crystalline size of nanoparticles was calculated by scherrer equation provided that the nanocrystelline size is less than 100nm. Where d is the average crystalline dimension perpendicular to the reflecting phase. λ is x-ray wavelength. K is constant close to units that is related both to the crystalline shape and to the way is defined i.e. ratio of the peak area to peak maximum. The macrostructure and morphology of sintered powder at 700^0c were characterized at room temperature by scanning electron microscopy (SEM) (HIT ACHI JAPAN) IR spectra have been recorded in the range at room temperature by the IR spectroscopy (Bruker).

Experimental Procedure:-

Chemical Reaction: for x=0.2

$$.5\ Mg(NO_3)_2.6H_2O + .3\ Zn(NO3)_2xH_2O + 0.2\ Ni(NO_3)_2.6H_2O + 2Fe(NO_3)7\ H_2O + 3\ C_6H_8O_7 \xrightarrow{\triangle} Mg_{.5}Zn_{.3}Ni_{0.2}\ Fe_2O_4$$

100^oC for 4-4.5 hrs

Chemical Reaction: for x=0.4

$$3\ Mg(NO_3)_2.6H_2O + .3\ Zn(NO3)_2xH_2O + 0.4\ Ni(NO_3)_2.6H_2O + 2\ Fe(NO_3)7\ H_2O + 3\ C_6H_8O_7 \xrightarrow{\triangle} Mg_{.3}Zn_{.3}Ni_{0.\ 4}Fe_2\ O_4$$

100^oC for 4-4.5 hrs

Chemical Reaction: for x=0.6

$$.1\ Mg(NO_3)_2.6H_2O + .3\ Zn(NO3)_2xH_2O + 0.6\ Ni(NO_3)_2.6H_2O + 2\ Fe(NO_3)7\ H_2O + 3\ C_6H_8O_7 \xrightarrow{\triangle} Mg_{\ 0\ .1}Zn_{.3}Ni_{0.6}Fe_2O_4$$

100^oC for 4-4.5 hrs

Fig. A: Experimental chemical reaction procedure

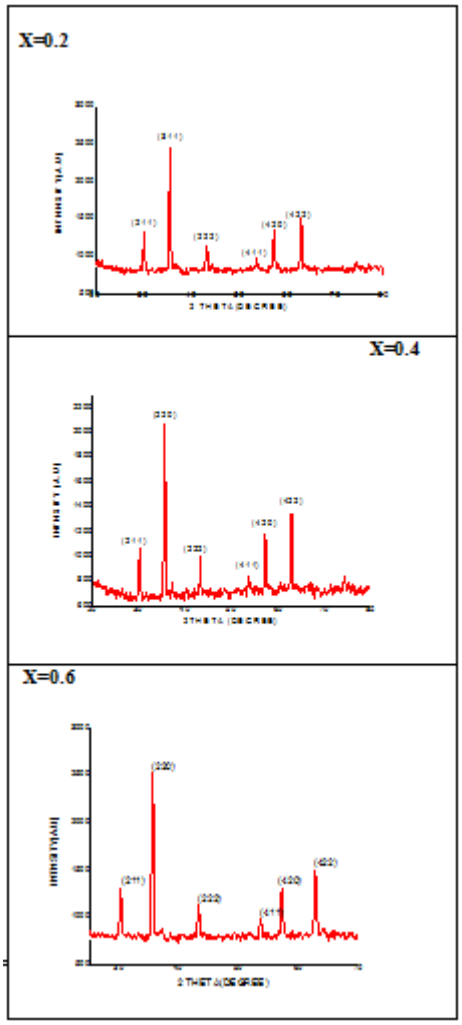


Fig1.-XRD patterns of of $Mg_{0.7-x}$ Ni_x $Zn_{0.3}$ nano ferrites at 700 ^{0}c

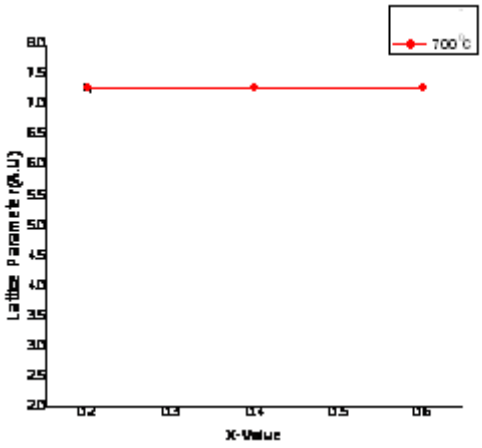


Fig. 3.Variation in the lattice parameter as function of X-Value at 700^0 c .

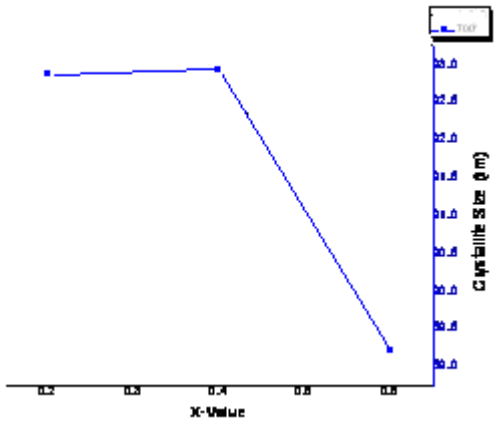


Fig.4 Crystallite size of sintered ($Mg_{0.7-x}$ Ni_X ,$Zn_{0.2}$) Fe_2O_4 ferrite with different Ni content

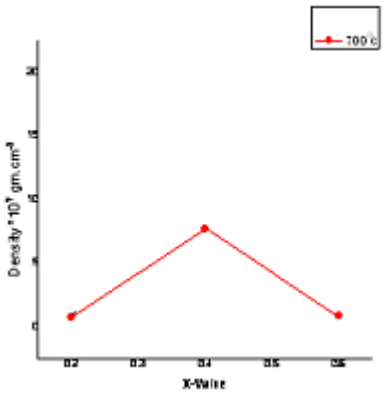


Fig.5 Variation of X-ray density with X-value of ($Mg_{0.7-x}$ Ni_X ,$Zn_{0.2}$) Fe_2O_4 ferrite with different

Result and discussion

Fig.1 shows the XRD paterns for the general formula ($Mg_{0.7-x}$ Ni_x $Zn_{0.3}$ Ferrite samples with various values of x at 700^0c. The phase formation behavior of $Mg_{0.7-x}$ Ni_x $Zn_{0.3,\ x}$=0.2, 0.4, 0.6) was studied by XRD. There was no metal oxide phase in the burnt powder. The crystalline size was calculated from Full Width at Half Maxima (FWHM) for all the peaks using Scherrer formula. $t = 0.9\lambda/\beta \cos\theta$

Where β= full width at half maximum, θ = Bragg's angle for the actual peak.

The crystalline size "t" of sintered powder was in the range 89.21 nm to 92.87nm. This shows that synthesized powder has nano-sized crystallites. All the bragg's angles of corresponding peaks in XRD pattern matches exactly with the characteristics of reflection peaks of Mg Ni Zn ferrites reported in JCPDS by Barakat, metal, J.Therm Anal 37.241 (1991)

Crystalline size(t), lattice parameter(a), & X-ray density(D_x) of sintered $Mg_{0.7-x}$ Ni_x $Zn_{0.3}$ Fe_2O_4 with different Mg content Fig. 3 shows lattice parameter remains same for x=0.2,0.4,0.6 . Fig. 4: shows as x value increased the crystal size increased and nearly remains same. Fig: 5 show X-ray density is maximum for x=0.4 for Mg Ni Zn samples sintered at 700^0c. The therotical density is the X-ray density (D_x) calculated by formula $D_x=ZM / Na^3$. Where Z is no. of molecules per unit cell. (Z=8), M is molecular weight, N is Avogadro's number. X-ray density decreases with increase in sintering temperature.

Table 1: lattice parameter Crystalline size and X-ray density of $Mg_{0.7-x}$ Ni_x $Zn_{0.3}$ Fe_2O_4 with different Mg content

Mg content X	Lattice parameter (a) (A.U)	Average crystalline size (t) nm	Density(D_X) $*10^7$ gm.cm^{-3}
	700^0c	700^0c	700^0c
0.2	7.28071	92.87	0.7476
0.4	7.2807	92.94	7.7694
0.6	7.28071	89.21	0.7928

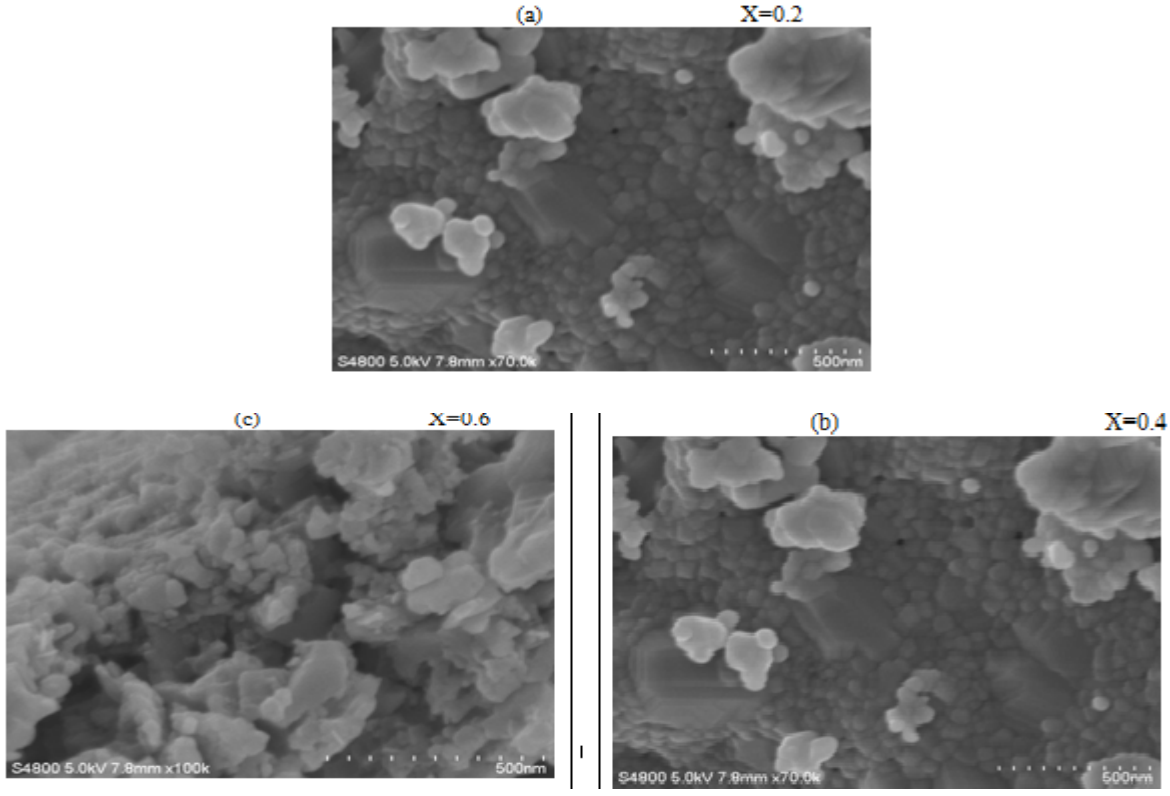
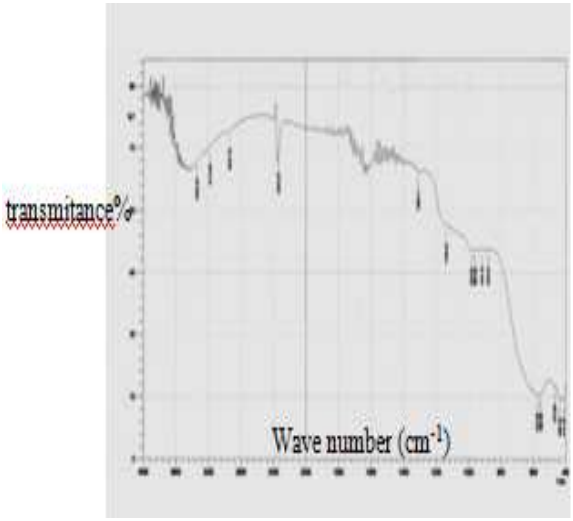


Fig.6: Microstructures of $Mg_{0.7-x}$ Ni_x $Zn_{0.3}$ Fe_2O_4 with various X- Values sintered at 700 ^{0}c

The structural morphology for different properties of $Mg_{0.7-x}$ Ni_x $Zn_{0.3}$ Fe_2O_4 nanoferrite is studied by using Scanning Electron Microscopy (SEM) Fig 6: represents SEM of Mg Ni Zn nanoferite powder calcinated at 700^0c for 2 hours. It is observed that majority of grains are nearly spherical in shape. (Journal of alloy and compound)

Infrared study of Mg Ni Zn

In the present study $Mg_{0.7-x}$ Ni_x $Zn_{0.2}$ Fe_20_4 has been synthesized using temperature at 700^0c using Sol- gel auto combustion method. The synthesis process is carried out by using analytical grade compounds, citric acid, ferric nitrate, magnesium nitrate, nickel nitrate and zinc nitrate. Ferric nitrate was used as a precursor and citric acid was used as reducing agent in reaction. The average particle size for two different reaction conditions was found in between 92.87nm and 89.21nm. The infrared curve of the sintered powder shows infrared peak at 555.52 cm-1 and 559.38 cm^{-1} for samples at 700^0c. The significant band for hydroxyl group and carbonyl group and nitrate group decrease in the final sample which clearly shows that citric acid and ferric nitrate gets consume during the course of reaction



Conclusion:

X-ray diffraction patterns confirm the formation of cubic spinal phase. The lattice parameters for 700^0c remain constant. The IR curve of the sintered powder shows IR peak at 555.52 cm^{-1} and 559.38cm^{-1}. IR curve confirms the ferrite formation. SEM shows that the majority of grains are nearly spherical in shape [8].

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"Cite this article"

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