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MORPHOLOGICAL AND ELECTRICAL STUDIES OF SPINEL FERRITE PREPARED AT DIFFERENT SINTERING TEMPERATURE

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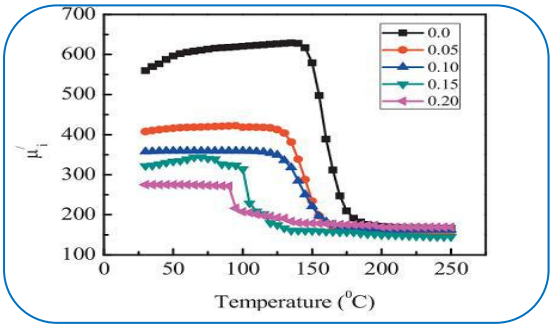
ABSTRACT :



High temperature properties in nanocomposite have received significant attention in the recent year due to its widespread application. Several synthesis methods have been used to prepared spinel ferrite nanoparticles. Sol-gel auto combustion technique have shown promising result in controlling the particle morphology and their size distribution, gives better homogeneity and gives high quality powder. In this work sol-gel autocombution method was used for preparation of mixed NZP powder using L-Ascorbic acid as fuel. The prepared powder is sintered at three temperatures 500^{0}C, 700^{0}C, and 900^{0}C for 6 hrs and its influence on morphological and electrical properties is studied and results obtained are presented in this paper.

KEYWORDS : *High temperature properties , Several synthesis methods.*

1. INTRODUCTION

The spinel ferrites with formula MFe_2O_4 are technologically important magnetic materials from the point of view of their potential applications and novel properties. Recently, there has been an increase interest in these magnetic nanoparticles because of their unique physiochemical, electrical, magnetic, dielectric and optical properties [1-4]. Many researchers have studied magnetic nanoparticle for various applications. The most conventional magnetic nanoparticles are iron oxide, γ-Fe_2O_3 and spinel ferrites. In the nano region spinel ferrite nanoparticles exhibit interesting, unusual and superior properties as compare to their bulk counterpart. Crystallite size and specific surface area of spinel ferrites are responsible for their superior properties. Nanoparticles of spinel ferrites have great potential for catalytic degradation of organic and inorganic pollutants, as a catalyst and as a sensor [5, 6]. Recently, they have also attracted considerably for biomedical applications such as contrast agents for magnetic resonance imaging (MRI), hyperthermia applications and in drug delivery system, microwave absorbance, magnetic fuel, Catalyst, multilayer chip inductor (MLCI), electromagnetic interference (EMI), suppression, gas sensing, transformer cores, antenna rods, inductors, recording heads etc. [7-11]. The high electrical resistivity, low eddy current and dielectric losses, moderate saturated magnetization and high Curie temperature are the important properties of Nickel ferrite. These properties are sensitive to various parameters like method of preparation, preparative parameters and conditions, nature of dopant and cation distribution. Due to their comparatively low losses at high frequencies, they are extensively used in specific applications such as switch mode power supply (SMPS). They are also used in high frequency circuits, high quality filters, read and write etc. [12-13].

Nickel ferrites are one of the versatile and technologically important soft ferrite materials due to their typical ferromagnetic properties, low conductivity and thus lower eddy current losses, high electrochemical stability, catalytic behavior, abundance in nature etc. Soft magnetic materials with their remarkable electrical, magnetic and optical properties have wide range of applications in various areas. Among the soft magnetic materials mixed spinel ferrites like Ni-Zn is of great importance because of their scientific and industrial application. Ni-Zn spinel ferrite has been extensively used in many electronic devices because of their moderate electrical, magnetic properties and good chemical stability.

The properties of the ferrite nanoparticles can be further tuned by controlling their shape, size and surface structure. Various techniques have been developed for the synthesis of ferrite nanoparticles. These method includes, sol-gel method, thermal decomposition, chemical co-precipitation, hydrothermal, mechanical milling etc. [14-18]. Among these methods sol-gel auto combustion method have shown promising results in controlling the particle morphology and their size distribution. The sol-gel auto combustion methods yields particles of nanometer dimension, gives better homogeneity and high quality powder. Various stabilizing agent/fuel such as citric acid, glycine, urea, polyethylene oxide, polyglycol etc. are reported to be used for the controlled growth, prevention of agglomeration and stabilization of nanoparticle. However, the properties of spinel ferrite nanoparticles are also dependent on sintering temperature, sintering time and sintering atmosphere. The sintering temperature affects the microstructure there by change in the properties.

2. EXPERIMENTAL

Synthesis of nanoparticles

Sol-gel auto combustion method is based on oxidation reduction process. The energy to form the ferrite nanoparticles of nanometer dimensions is provided by oxidation reduction process of chemical precursors and fuel used in the sol-gel auto combustion method. In the synthesis of Metal nitrates of constituent ions i.e. nickel nitrate, zinc nitrate and ferric nitrate of AR grade supplied by Merck were used as a raw materials. L-ascorbic acid was used as a fuel. The metal nitrate to fuel ratio was taken to be 1:3. For synthesize Ni-Zn ferrite of composition of $Ni_{0.65}Zn_{0.35}Fe_2O_4$ calculated amount of all nitrates and L-Ascorbic acid were first dissolved in minimum amount of distilled water and stirred for 30 minutes and then mixed together. This initial solution was highly acidic. Ammonia was added to adjust the pH of mixed solution at 8 .The mixed solution was kept onto a hot plate with continuous stirring at 750 to get a dense sticky gel. After gel formation, the temperature was increased to 110 ^{0}C for the dehydration process. The temperature was then increased rapidly and when it reached approximately to 120 ^{0}C, large amount of gases CO_2, H_2O, N_2) were liberated, and a dark brown ferrite power was produced through the combustion process. The prepared powder of Ni-Zn spinel ferrite is sintered at different temperature viz. 500°C, 700°C and 900°C and effect of sintering temperature on morphology and electrical properties of Ni-Zn ferrites was studied.

3. CHARACTERIZATION

3.1 Morphological characterization

The surface morphological studies were carried out using scanning electron microscopy using JEOL JSM-6360 scanning electron microscope (SEM).

3.2 Electrical measurements

The electrical properties of the $Ni_{0.65}Zn_{0.35}Fe_2O_4$ at temperature 500°C, 700°C and 900°C were measured by two probe techniques. The prepared nanopowders were mixed with polyvinyl alcohol and pressed into cylindrical pellets under 5 ton pressures, after that pellet was sintered at 500°C for 2 h. By applying the silver paste on the surface of the pellet, it was used for DC resistivity measurements using the two probe technique. The measurements were performed in the temperature range 300-800 K. The temperature of the sample was sensed by chromel-alumel thermocouple with an accuracy of ± 5 K.

4. RESULTS AND DISCUSSIONS

4.1 Morphological characterizations

Scanning Electron Microscopy (SEM)

SEM micrographs on Ni-Zn samples sintered with 500°C, 700°C and 900°C are shown in Fig 4.1.(a-c) SEM analysis confirms the formation of nanostructured ferrite. Uniform grown grain with agglomeration on the particles is observed in the SEM images. The remarkable growth in particle size is observed with increasing sintering temperature using SEM analysis. The grain size and surface area were calculated and their values are presented in Table 4.1

Energy Dispersive X-Ray Spectroscopy (EDAX) measurements as shown in Fig.4.2 (a-c) are carried out for the present samples to analyse the estimated stoichiometry. EDAX analysis confirms the stoichiometry proportions of the constituent ions as desired. The estimated and expected stoichiometry proportions of Ni-Zn and Fe is matched with each other as presented in Table 4.2

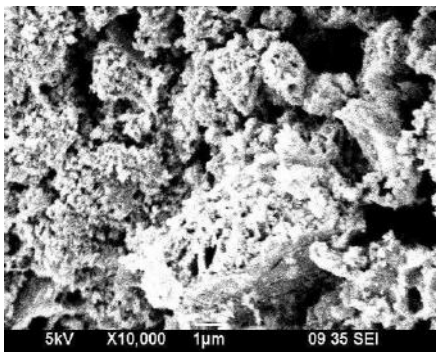


Figure 4.1 (a): SEM image of NZP nanoparticles for (500 °C)

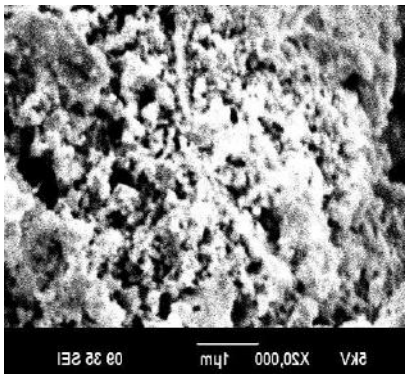


Figure 4.1 (b): SEM image of NZP nanoparticles for (700 °C)

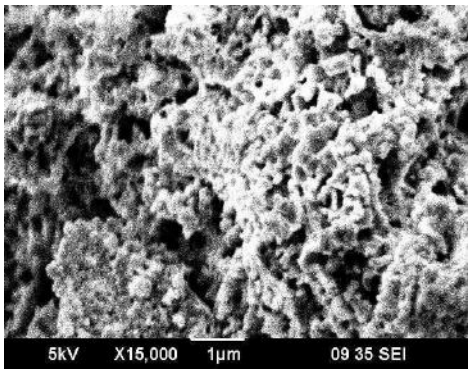


Figure 4.1 (c): SEM image of NZP nanoparticles for (700 °C

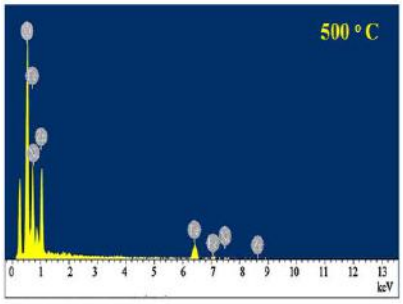


Fig. 4.2 (a): EDAX pattern of NZP nanoparticles for (500 °C

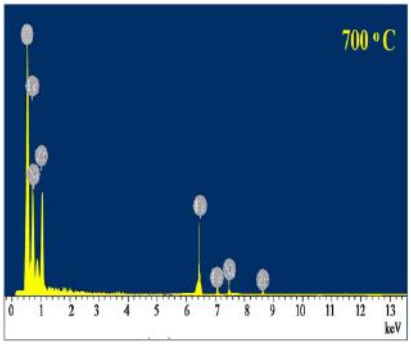


Fig. 4.2 (b): EDAX pattern of NZP nanoparticles for (700 °C)

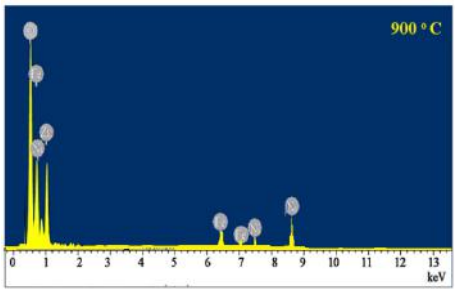


Fig. 4.2 (c): EDAX pattern of NZP nanoparticles for (900 °C)

4.2 Electrical Properties

D. C. Electrical resistivity measurements

The DC electrical resistivity studies of the prepared $Ni_{0.65}Zn_{0.35}Fe_2O_4$ nanoparticles sintered at 500 °C, 700 °C and 900 °C ferrite samples were carried out in the temperature range of 500 K – 715 K. The resistivity for each sample at the same temperature was calculated and logarithm of the resistivity (*log ρ*) was plotted as a function of reciprocal of temperature (*1000/T*). Fig.4.3 shows the variation of logarithm of resistivity as a function of reciprocal of temperature. It is evident from figure 4.3 that the resistivity decreases with increasing temperature obeying Arrhenius relation [19] .This confirms the semiconducting behaviour of all the prepared Ni-Zn spinel ferrite samples under investigation. DC electrical resistivity of the samples was found to be decreased with the increase in sintering temperature.

The slope of the linear plots drawn of log (ρ) v/s 1000/T gives the activation energy of the samples. The activation energy was calculated for each sample and the values are tabulated in the Table 4.3 along with the other parameters. Activation energy is found to decrease from 0.73 eV to 0.28 eV with increases in sintering temperature from 500 °C to 900 °C. The decrease in activation energy may be due to creation of smaller number of oxygen vacancies. The graphs drawn in the Fig.4.3 shows slope at particular temperature indicating that sample undergoes change from ferrimagnetism to paramagnetism.The temperature at which changes its state may correspond to Curie temperature. The similar behaviour of the present samples is analogous to that of reported in the literature [20.]

Sintering temp. maintain at	Grain Size (nm)SEM	Specific Surface Area (m^2/g)
500 ° C	74	14.93
700 ° C	79	13.62
900 ° C	71	15.55

Table 4.1 Grain size (G), specific surface area (S) from typical scanning electron microscopy (SEM) image for $Ni_{0.65}Zn_{0.35}Fe_2O_4$ nanoparticles (500 ° C, 700 ° C and 900 ° C)

Sintering temp. maintain at	Ni^{2+} (%)	Zn^{2+} (%)	Fe^{3+} (%)	O^{2-} (%)
500 ° C	62.42	17.93	14.27	5.38
700 ° C	59.74	18.93	13.99	7.34
900 ° C	61.71	19.00	12.55	6.74

Table 4.2 Elemental percentage of $Ni_{0.65}Zn_{0.35}Fe_2O_4$ nanoparticles (500 ° C, 700 ° C and 900 ° C)

Sintering temp. maintain at	Activation Energy		
	E_P (eV)	E_f (eV)	ΔE (eV)
500 ° C	4.21	3.48	0.73
700 ° C	3.57	3.23	0.34
900 ° C	3.25	2.97	0.28

Table 4.3 Activation energy (E_P, E_f and ΔE) of $Ni_{0.65}Zn_{0.35}Fe_2O_4$ nanoparticles for (500 ° C, 700 ° C and 900 ° C)

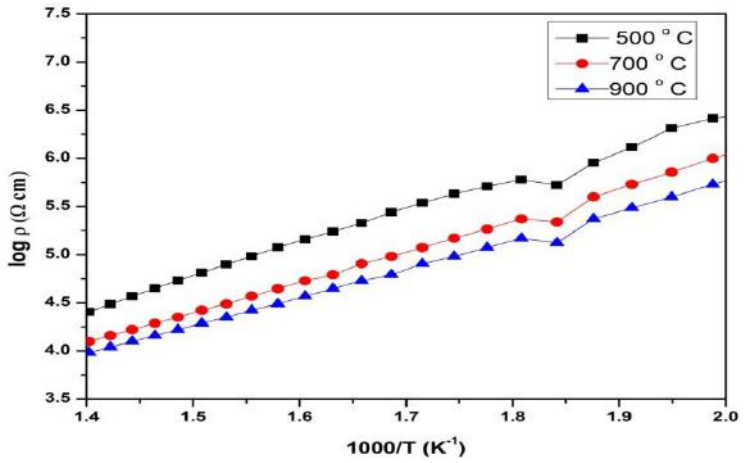


Fig.4.3 Variation of D.C. resistivity with reciprocal of temperature of $Ni_{0.65}Zn_{0.35}Fe_2O_4$ nanoparticles for (500 °C, 700 °C and 900 °C

5. CONCLUSIONS

The nanocrystalline Ni0.65Zn0.35Fe2O4 ferrite system for different sintering temperature viz. 500 °C, 700 °C and 900 °C was successfully synthesized via sol-gel auto combustion technique. The average grain size determined from scanning electron microscopy technique is of the order of 71-79 nm.DC electrical resistivity decreases with increase in temperature obeying Arrhenius relation. The DC electrical resistivity measurements show that the resistivity decreases as sintering temperature increases.

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