CHEMICAL SYNTHESIS ROUTE AND ANALYSIS OF DIELECTRIC PROPERTY OF NANOCRystALLINE Ni$_{0.6}$ Zn$_{0.4}$ (Fe$_2$O$_4$) SPINEL FERRITE

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ABSTRACT

The powders of nano crystalline Ni$_{0.6}$ Zn$_{0.4}$ (Fe$_2$O$_4$) was synthesized by using chemical co-precipitation method and followed by annealing temperature at 100$^\circ$C, 600$^\circ$C and 900$^\circ$C. X-ray powder diffractometry (XRD), Scanning Electron Microscopy (SEM) and Fourier Transform Infrared Spectroscopy (FTIR) were carried out to investigate structural and chemical aspects of Ni$_{0.6}$ Zn$_{0.4}$ ferrite. The cubic spinal structure in single phase has been confirmed by X ray diffraction. The average crystallite size was calculated from the line broadening in XRD pattern also observed that the size of the particles was increased with sintering temperature. The surface morphology of the samples was studied using Scanning Electron Microscopy (SEM) technique. The IR Spectra for the samples measured in the range of 4000-400 cm$^{-1}$ exhibit symmetric stretching mode of vibration of tetrahedral and octahedral sites at 669.7 cm$^{-1}$ and 545.6 cm$^{-1}$. The dielectric property of the Ni$_{0.6}$ Zn$_{0.4}$ (Fe$_2$O$_4$) was also studied for different temperatures.

Key Words: Co-precipitation, XRD, SEM, FTIR, Ferrite

INTRODUCTION

Recent interest in the study of several spinel-type ferrites has focused on the development of nanosized particles at low temperatures by different chemical synthesize techniques in view of the potential applications of these nanosized magnetic materials in different technological areas, as well as studies of the dielectric and magnetic properties on nano ferrite materials$^{1,2}$. Research on the application of ferrite for protecting the natural environment is currently being actively investigated for the washer disposal method for factory drains, heat decomposition of CO$_2$ using a mixture of carbon and ferrite at 300-700$^\circ$C, transformation of solar energy to hydrogen energy using ferrites as catalysts and heat decomposition of NOx gas using spinel ferrite.

Among the different spinel type ferrite, nickel zinc ferrites are the most versatile magnetic materials for general use. They have many applications in low and high frequency devices and play a useful role in many technological applications such as microwave devices, power transformers in electronics, rod antennas, read/write heads for high speed digital tape etc. Due to their high resistivity, low dielectric losses, mechanical hardness, high curie temperature, chemical stability$^{3-5}$ and small eddy current loss when operating at high frequencies (10-500MHz)$^6$. Nickel zinc ferrite is a mixed spinel in which the tetrahedral (A) site are occupied by Zn$^{2+}$ and Fe$^{3+}$ ions and the octahedral (B) site are occupied by Ni$^{2+}$ and Fe$^{3+}$ ions in the spinal formula AB$_2$O$_4$. The distribution of the various ions in tetrahedral and octahedral sites is different when the ferrite is synthesized at low temperatures with the particle size in a nanometric scale.$^7$
It is also known that the physical properties of the ferrites are very sensitive to the method of synthesis. The selection of an appropriate process is therefore the key to obtain good quality ferrites. Various synthesis processes have been used for preparing nano sized ferrite particles. These includes sol-gel, hydrothermal, chemical coprecipitation, sonochemical reactions, mechanical ball milling etc. Of all these process chemical coprecipitation seems to be most convenient method for the synthesis of nanomagnetic particles as large yield rate. It is very simple and has better control over crystalline size and other properties of the materials. The dielectric behavior of the samples were analyzed and reported.

**MATERIAL AND METHODS**

Ferrite nano particles were prepared by chemical co precipitation route using metal chlorides such as NiCl₂,6H₂O, ZnCl₂,2H₂O, FeCl₃,6H₂O. Metal chlorides with stoichiometric calculation of \( \text{Ni}_{0.6-\delta}\text{Zn}_{0.4}(\text{Fe}_2\text{O}_4) \) was dissolved in minimum volume of distilled water with constant stirring by a magnetic stirrer, until a clear solution is obtained. The precipitating reagent is prepared in distilled water with NaOH by taking their appropriate weights depending upon amount of metal chlorides taken for co precipitation of ferrite precursor. The precipitating reagent is added drop wise in to the metal solutions contained in a beaker with constant mixing until co-precipitation occurs. The precipitates were thoroughly washed several times with distilled water to remove salt residues and other impurities. The product was dried in a muffle furnace at a temperature of 100°C for overnight to remove water contents. The resultant dried product is powdered by using agate mortar and pestle to have very fine particles. The powder was sintered at 100°C, 600°C and 900°C. The crystallite size was determined from the X-ray diffraction (XRD) data. XRD data were taken at room temperature using CuKα (\( λ = 1.5406\text{Å} \)) radiation. The scanning electron micrograph was recorded by using Leica Stereo Scan 440 scanning electron microscope. Infrared spectroscopic analysis using KBr pellets were carried out at the range of 4000-400 cm\(^{-1}\) in a Perkin-Elmer FTIR RXI spectrometer. The dielectric property of the sample was tested using LCR meter in the frequency range 50Hz – 200 KHz.

**RESULTS AND DISCUSSION**

The X-ray diffraction patterns confirm the single spinel structure of \( \text{Ni}_{0.6-\delta}\text{Zn}_{0.4}(\text{Fe}_2\text{O}_4) \) prepared via the coprecipitation method is shown Fig.1. The entire peaks match well with the standard one with no extra line corresponding to the other phase. Table 1 shows the particle size(t), lattice constant (a) and X-ray density (d) of the sintered samples. Fig. 2 shows the effect of sintering temperature on particle size of the synthesized samples. The average grain size is determined using the Scherrer’s formula. The particle size of the samples increase from 20.75nm to 27.85nm with the increase of temperature. The XRD pattern shows peak intensity for the crystalline phase increased and the peak width decreased with increase in sintering temperature, which represents the well crystalline single phase. The sharper peaks indicate the crystalline size increases with sintering temperature. IR spectra analyses of the sintered samples in the range 4000-400 cm\(^{-1}\) are shown in Fig. 3. The characteristics bands at intensities H 3400 cm\(^{-1}\), H 1599 cm\(^{-1}\) and H 1380 cm\(^{-1}\) are due to O-H stretching vibration interacting through bonds. The intensities for the bonds corresponding O-H stretching vibration decreases drastically when sintering temperature is increased. This decrease is due to the loss of the residual water in the samples. The stretchy vibration of tetrahedral size of nickel zinc ferrite at 669.7cm\(^{-1}\) was appeared, similarly the octahedral site is appeared at 545.6 cm\(^{-1}\).

Fig. 4 shows SEM image of \( \text{Ni}_{0.6-\delta}\text{Zn}_{0.4}(\text{Fe}_2\text{O}_4) \) sample sintered at 600°C. SEM image aggregates to non uniform particles having orbicular shapes with porous nature. This might because of re-evaporation of zinc atoms during the sintering.

Fig.5 Shows the variation of dielectric constant with respect to applied frequency for different annealing temperatures. It reveals that dielectric constant decreases suddenly with an increase in frequency and maintain a steady state or constant at higher frequencies. This trend is generally maintained for spinel ferrites. The annealing temperature of the \( \text{Ni}_{0.6-\delta}\text{Zn}_{0.4}(\text{Fe}_2\text{O}_4) \) nano particles has no pronounced effect on the
dielectric constant in high frequency range but significantly decreases the dielectric constant in high annealing temperature. This type property behavior was observed for number of ferrites. However, as the frequency of the applied field is increased, the electrons reverse their direction of motion more often. Therefore with an increase of frequency in the applied field the dielectric constant decreases.

**Fig. 6** shows the variation in the dielectric loss factor of Ni-Zn ferrite sintered at various temperatures. The values of the dielectric loss decreases with an increase of the frequencies. Both the dielectric constant and dielectric loss decrease as the frequency increases indicating the normal behavior of ferrites. This decrease is rapid at the lower frequencies and become slow at the higher frequencies. This change takes place when the jumping frequencies of electric charge carriers cannot follow the alteration of applied AC electric field beyond a certain critical frequencies.

![X-ray diffraction patterns of the sintered samples.](image)

**Table 1 : Crystal size (t), Lattice constant (a) and X-ray density (d<sub>x</sub>) of the synthesized samples**

<table>
<thead>
<tr>
<th>Sintering Temp. (°C)</th>
<th>Particle size (t) (nm)</th>
<th>Lattice constant (a) (Å)</th>
<th>X-ray density (d&lt;sub&gt;x&lt;/sub&gt;) (g.cm&lt;sup&gt;-3&lt;/sup&gt;)</th>
</tr>
</thead>
<tbody>
<tr>
<td>100</td>
<td>20.75</td>
<td>8.351</td>
<td>6.841</td>
</tr>
<tr>
<td>600</td>
<td>21.34</td>
<td>8.641</td>
<td>6.139</td>
</tr>
<tr>
<td>900</td>
<td>27.85</td>
<td>8.658</td>
<td>6.175</td>
</tr>
</tbody>
</table>
Fig. 2: Variation of particle size with temperature

Fig. 3: IR spectra of the sintered samples

Fig. 4: Scanning electron micrograph of the sintered sample
Fig. 5: Variation of dielectric constant with frequency

CONCLUSION

This work has successfully synthesized Ni-Zn ferrite nanoparticles by chemical co precipitation technique. The characterization studies such as XRD SEM confirm the nanocrystalline nature of the particles. The particle size of the synthesized Ni-Zn ferrite is below 28nm. An FTIR spectrum of the samples confirms the intensities for the bands corresponding to tetrahedral and octahedral sites. The dielectric constant and the value of dissipation factors were decreases with an increase of applied frequency.

REFERENCES


