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Effect of temperature on Zn-Cu mixed ferrite Nano particles

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Abstract

Nano-crystalline Zn-Cu mixed ferrite is synthesized using co-precipitation method. Zinc nitrate [$Zn(NO_3)_2.6H_2O$], copper nitrate [$Cu(NO_3)_2.3H_2O$], ferric nitrate [$Fe(NO_3)_3.9H_2O$] along with sodium hydroxide [NaOH] are used as raw materials. As prepared and sintered Zn-Cu mixed ferrite powders are subjected to XRD analysis to calculate average particle size. The consistency of Nano size of the particle is analyzed using TEM micrograph. The FT-IR spectrum of the sample is recorded at different sintering temperatures. The magnetic measurements of Zn-Cu mixed Ferrite nano particles are carried out using Vibrating Sample Magnetometer (VSM).

Keywords: co-precipitation, ferrite, micro graph, nano particles, nano size, sintered, Vibrating Sample Magnetometer

INTRODUCTION

The synthesis of spinel ferrite nano particles has been investigated intensively in recent years because of their potential applications in high density magnetic recording microwave devices and magnetic fluids (Goldman, 1990; Berkovsky, 1993). The interesting physical and chemical properties of spinel ferrites arise from the distribution of transition metal cations having various oxidation states, among the available tetrahedral and octahedral sites (Romeijn, 1953; Blasse, 1964; Mathew *et al.*, 2004). Various preparation techniques such as sol-gel methods (Dos *et al.*, 2001; Chae *et al.*, 2002), citrate precursor techniques (Prasad, *et al.*, 1998; Panda, 2003), electro chemical synthesis (Sartale *et al.*, 2002), combustion methods (Yan *et al., 1999)*, Solid state reaction (Godinho *et al.*, 2002) and mechanically alloying (Ding *et al.*, 1994; Ding *et al.*, 1997; Shi *et al.*, 2002) are used to produce ferrite nanoparticles. Of all these techniques, chemical co-precipitation seems to be the most convenient for the synthesis of nano particles because of its simplicity and better control over crystallite size and other properties of the materials.

The synthesis of nano crystalline Zn-Cu mixed ferrites by co-precipitation method are reported. The structural characteristics of Zn-$CuFe_2O_4$ samples are analyzed by XRD and TEM. The FT-IR spectrum of the sample is recorded at different sintering temperatures. The magnetic measurements of Zn-Cu mixed ferrite nanoparticles are carried out using Vibrating Sample magnetometer(VSM).

METHODS

Ferrite nano particles $Zn_{(1-x)}Cu_xFe_2O_4$ (x=0.2) were prepared by chemical co-precipitation method. The desired composition was obtained by taking stoichiometric amounts of zinc nitrate[$Zn(NO_3)_2.6H_2O$], copper nitrate [$Cu(NO_3)_2.3H_2O$] and ferric nitrate [$Fe(NO_3)_3.9H_2O$] dissolved in distilled water. The neutralization was carried out with sodium hydroxide solution and the reaction temperature was maintained at 60°C. The pH of the solution was maintained at 8 and stirred for 2hrs. The precipitate was thoroughly washed with distilled water until their washing are free from impurities. The product was dried to a temperature of 100°C to remove water contents. The dried powder was mixed homogeneously and are sintered at 130°C, 600°C and 900°C.

The samples were characterized by X-ray diffraction(XRD) using Cu Kα radiation to confirm the crystal structure and to estimate the average particle

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size. Transmission Electron Micrographs (TEM) were recorded to analyze the consistent of particle size with XRD.

RESULTS AND DISCUSSION

IR Spectral Analysis

The IR transmission spectra for Zn-Cu mixed ferrites sintered at 130°C, 600°C and 900°C were recorded in the range of 4000-400cm^{-1} and are shown in Fig.1. The FT-IR spectra showed main absorption bands at ~560cm^{-1} corresponding to the vibration modes of all spinel compounds. The ferrite can be considered as continuously bonded crystals through ionic, covalent or vander waals force to the nearest neighbours. In ferrite the metal ions can be situated in two different sublattices, namely tetrahedral (A-sites) and octahedral (B-sites) according to the geometric configuration of the oxygen nearest neighbour. The band around 560 cm^{-1} is attributed to stretching mode of tetrahedral complexes (Selvan *et al.*, 2003). The absorption broad band at ~1300 cm^{-1} represents the stretching mode of H_2O molecules and OH groups. The band around ~1600 cm^{-1} corresponds to the bending mode of H_2O molecules (Huang *et al.*, 2006).

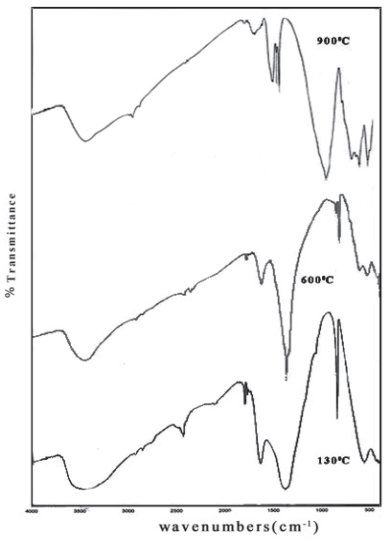


Figure 1. FTIR spectra for $Zn_{0.8}Cu_{0.2}Fe_2O_4$ nanoparticles synthesized at different temperatures (130°C, 600°C and 900°C)

XRD

X-ray diffraction pattern were obtained with X-ray powder diffractometer using Cu Kα radiation (λ=1.54A°) and graphite crystal monochromator. The powder diffraction patterns of Zn-Cu ferrites are reproduced in Fig.2. The spinel peaks corresponding to [220], [311], [222], [400], [422], [511], [440] planes can be seen clearly confirm the formation of normal spinel structure. The lattice parameter is also estimated. The particle size calculated using Scherrer formula and found to be 20nm-35nm.

Particle size and morphology of the powder were predicted from TEM images. The TEM images of the prepared sample are shown in Fig.3. The non- agglomerated well crystalline nature of ZnCu ferrite

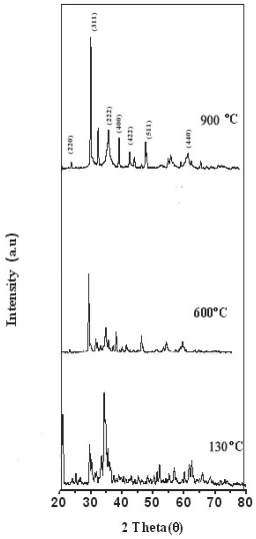


Figure 2. X-ray diffraction patterns for $Zn_{0.8}Cu_{0.2}Fe_2O_4$ nanoparticles synthesized at different temperatures (130°C, 600°C and 900°C)

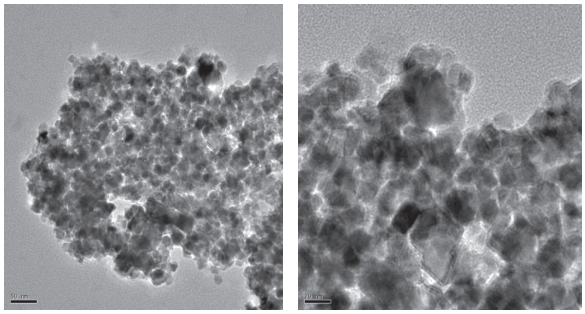


Figure 3. Transmission Electron Microscopy image for $Zn_{0.8}Cu_{0.2}Fe_2O_4$ nanoparticles

nanoparticles were observed. The crystallinity of the nanoparticles are also observed as very nearly uniform shapes and dimensions.

Magnetization studies using VSM

Variation of the magnetic moments with applied field are shown in Figures(4-6). Magnetic properties of ferrites have been explained by Neel (1948), who postulated that magnetic moments of ferrites are a sum of magnetic moments of individual sublattices. In the ferro spinels these are sublattice A consisting of cations in tetrahedral positions and sublattice B with cations in octahedral positions. Exchange interaction between electrons of ions in these sublattices has different value.

Usually interaction between magnetic ions of sublattices A and B (A-B interaction) is the strongest. A-A interaction is almost ten times weaker and B-B interaction is the weakest. For higher Zinc concentration (x >0.4-0.5) ferrites become normal spinel. Higher the temperature, greater is the relative effect of the A-B exchange weakening due to the thermal fluctuations, resulting in decreased magnetization (Callen *et al.*, 1956). As in this case the maximum saturation magnetization (M_s) of 64.03E^{-3} emu is obtained for 130^{o}C and found to decrease with increasing annealing temperature. These results also confirm that the retentivity increases with increase in annealing temperature. In ferrites, the variation in M_s has been attributed to both surface spin effects and cation distribution between the A and B sites (Upadhyay *et al.*, 1949; Rath *et al.*, 2002) surface effects lead to the formation of a magnetic dead layer resulting in a decrease in M_s.

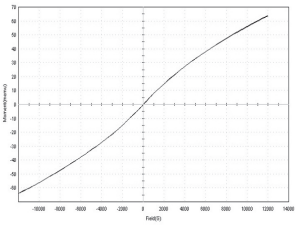


Figure 4

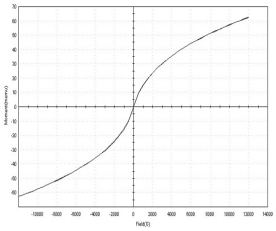
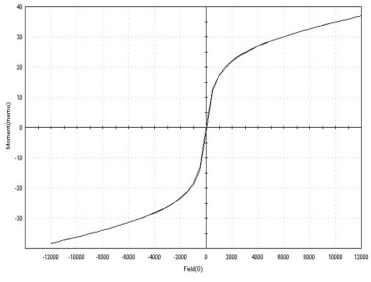


Figure 5



Figures (4-6). Vibrating Samples Magnetometer measurement results for $Zn_{0.8}Cu_{0.2}Fe_2O_4$ nanoparticles synthesized at different temperatures (130^{o}C, 600^{o}C and 900^{o}C)

The coercivity decreases initially and increases after annealing. This trend basically accompanied with the inverse relation between the coercivity H_c and the Saturation Magnetization. (i.e)

$$H_c \alpha k(M_o M_s)^{-1}$$

Where k is the magneto crystalline anisotropy constant (Yan Li *et al.*, 2007). The increase in the coercivity with increase in temperature may be due to the donation of Zn^{2+}ion in octahedral sites and it directs to the increase of the magneto crystalline anisotropy.

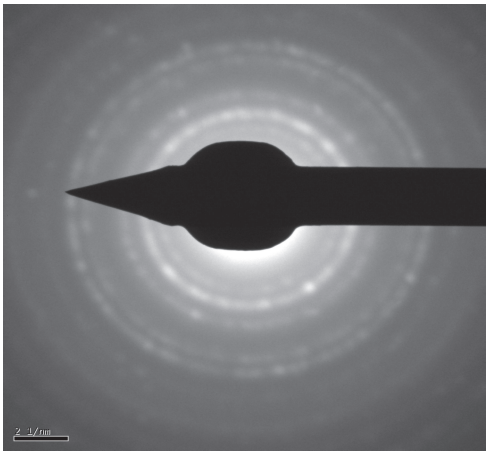


Figure 7. Selected Area Electron Diffraction (SAED) pattern for $Zn_{0.8}Cu_{0.2}Fe_2O_4$ nanoparticles

Fig.7 depicts the Selected Area Electron Diffraction (SAED) pattern of the powder. The observed pattern confirms the crystalline nature of the nano particles as observed by XRD.

CONCLUSION

$Zn_{(1-x)}Cu_xFe_2O_4$ nanoparticles are successfully prepared at low temperature by co-precipitation method. The FT-IR spectra show main absoption bands at ~560cm^{-1} corresponding to the vibration modes of all spinel compounds. XRD pattern shows crystalline nature for the sample.VSM studies suggest the saturation magnetization decreases linearly. It also reveals that the coercivity decreases and increases with increase in annealing temperature.

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