## EFFECT OF MULTIWALLED CARBON NANOTUBES ON THE STRUCTURAL AND MAGNETIC PROPERTIES OF Mn-Zn FERRITE

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Nano sized  $Mn_{0.5}Zn_{0.5}Fe_2O_4$  ferrite particles were synthesized by co-precipitation method. The composite of  $Mn_{0.5}Zn_{0.5}Fe_2O_4$  and multiwalled carbon nanotube have been successfully formed by the solid state reaction method. Multiwalled carbon nanotubes (MWCNTs) are substituted in the soft ferrite  $Mn_{0.5}Zn_{0.5}Fe_2O_4$ , with weight percent ratio of 1%, 5% and 9%. The effect of MWCNTs on the Structural, Thermal and Magnetic properties of Mn-Zn ferrites is reported. The X-ray diffraction analysis of sintered powder 1000°C reveals the ferrite possesses spinel face centered cubic structure. Structural, Thermal analysis of the MWCNTs and Mn-Zn ferrite size is observed by SEM ranging from 25nm to 40nm. The magnetic properties are measured by using the Physical property measurements (PPMS) technique. The Fourier transform infrared spectroscopy is used to detect the presence of the metallic compounds in the ferrite sample. The saturation magnetization of the  $Mn_{0.5}Zn_{0.5}Fe_2O_4/MWCNTs$  increases with the increase of MCNTs concentration. The small value of the coercivity indicates that the material is super paramagnetic.

(Received May 14, 2015; Accepted December 7, 2015)

*Keywords:* Multiwalled carbon nanotubes (MWCNTs); Physical property measurements (PPMS); Fourier transform infrared (FTIR) spectroscopy; X-ray diffraction.

## **1. Introduction**

Manganese-Zinc (Mn-Zn) elements are important in many high frequency power electronics and magnetic applications as a consequence of their high magnetic permeability and electrical resistivity. The concentration of ferrous and ferric ions and their distribution between the tetrahedral and the octahedral sub-lattices, play a critical role in determining their magnetic and electrical properties [1]. The use of electronic products and telecommunication equipment's has been increased with the advancement of electric and electronic industry as a consequence the problem of electromagnetic interference (EMI) has attained a special attention because it reduces the life time, competence of the instruments and also affects the safety operation of many electromagnetic destruction. Recently, extensive research has been done for the development of new microwave shielding materials that have high efficiency, light weight and a high durability. The composite based on polymers like ferrite/polymer composites, metal/polymer composites, epoxy-PANI/ ferrite composite and Single walled carbon nanotubes (SWCNTs) epoxy composites are suitable for microwave absorption [2-4].

Zinc ferrite has long been the subject of studying among the spinel ferrites, because it possesses unique properties such as chemical and thermal stability and the magnetic properties

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depends mostly on particle size [5]. Soft magnetic ferrites are used as cores in modern electronic components such as recording heads, filters, switching power supply transformers, amplifiers, etc. Mn-Zn ferrites attracted much attention due to a wide range of relative magnetic permeability value (from  $10^3$  to  $10^4$ ) and low magnetic losses as well as increased thermal stability, high saturation magnetic flux density at high temperatures (Bs>0.4T at 370K) and a relatively high curie temperature [6, 7], operating frequency is usually in the range of 1 KHz to 1MHz but in some applications the frequency in GHz range is used. Furthermore, excellent corrosion resistance and chemical stability enable them to be applied in extreme conditions.

Recently, a variety of preparation routes have been examined for Mn-Zn ferrite production; mechano-chemical processing [8, 9], Chemical co-precipitation method [10], sol-gel [11], or micro emulsion [12]. This paper deals with Mn-Zn ferrites prepared by co-precipitation technique. The spinel ferrites (MFe<sub>2</sub>O<sub>4</sub>, where M= Ni, Co,Zn, Mn, Cd, etc) are particularly important magnetic material due to their use in electromagnetic interference (EMI) in the MHz range like TV ghost suppressor [13-16]. The Mn-Zn spinel ferrites have preference over other ferrites due to their high initial permeability, high saturation magnetization, high resistivity, low eddy current losses, low hysteresis and relatively high Curie temperature [17, 18]. Mn-Zn ferrites have spinel structure with Fe ions at both tetrahedral (A-site) and octahedral site (B-site) while  $Mn^{2+}$  and  $Zn^{2+}$  ions occupy tetrahedral (A-site). The introduction of different metal cations concentration into the soft ferrites, leads to change in electric and magnetic properties of the material considerably [19]. Requirements for magnetic cores used in switching power supply transformers include soft magnetism, easy magnetization with a small external magnetic field, and low loss. Magnetic materials are classified into two groups, metallic materials and oxide materials. The electric resistance of the metallic materials is generally lower, driving the transformer of a switching power supply causes large eddy current loss at high frequencies. In order to suppress loss Mn-Zn ferrites are used in the transformer rather than metallic materials. The drawback of Mn-Zn ferrites is their saturation magnetic flux density which is lower than those in metallic soft materials. This means that a larger volume of ferrite core is required to produce the same amount of magnetic flux as metallic cores produce. Furthermore, the Curie temperature of Mn-Zn ferrite is also lower, being typically less than 250°C. The numerous researches for EMI shielding have shown that the multiwalled carbon nanotube (MCNT) would be applied as the EMI shielding material due to its exceptional mechanical, electrical and thermal properties [20-23]. Applications range of carbon nanotube composite is from electronics to aerospace sectors, such as electrostatic dissipation [24], multilayer printed circuits [25] and transparent conductive coating [26]. The multifunctional composites, CNT based magnetic composites have been emerging as an interesting area for the researchers due to their exceptional electromagnetic properties and potential application in magnetic data storage, inks for xerography, toners, magnetically guided drug delivery systems and magnetic resonance imaging. The aim of making the ferrite and carbon nanotube composite is to develop cheap and easy to process conductive material for future applications. In the present paper we have studied the structural, thermal and magnetic properties of Mn-Zn multiwalled carbon nanotubes composites.

### 2. Experimental method and calculations

#### **2.1 Materials**

All the chemical re agents  $Zn(NO_3)_2.6H_2O$ ,  $Mn(NO_3)_2.6H_2O$  and anhydrous ferric nitrate  $Fe(No_3)_3.9H_2O$  of Sigma Aldrich are taken while the hydrochloric acid HCl, Nitric acid HNO<sub>3</sub> are of Merck co. Germany. These acids are used to purify the MWCNTs. The length and diameter of the MWCNTs was 100µm and 20nm, respectively.

#### 2.2 Synthesis method

Nano crystalline ferrites  $Mn_{0.5}Zn_{0.5}Fe_2O_4$  are prepared by co-precipitation method. The desired composition is obtained by taking stoichiometric amount of Zn (NO<sub>3</sub>)<sub>2</sub>.6H<sub>2</sub>O, Mn (NO<sub>3</sub>)<sub>2</sub>.6H<sub>2</sub>O and Fe (NO<sub>3</sub>)<sub>3</sub>.9H<sub>2</sub>O are dissolved in deionized water. On adding the ammonia solution the pH of the solution becomes10 and it becomes neutral after stirring 2hrs. Put the

solution in a sonicater for 20 minutes then place it for a while in the ultrasonic bath so that precipitate will settle down then filter it with a filter paper. Wash the precipitate with deionized water until it becomes free of impurities. The product is dried to remove water contents. The dried powder is sintered at 1000°C and mixed with small multiwall carbon nanotubes with a weight percentage ratio of 1%, 5% and 9%. Several strategies have already been developed over the last decade to obtain MWCNTs as a pure as possible, and to minimize the influence of impurities on the performance of composites. Impurities can indeed affect the batch to batch reproducibility of the results, and accordingly influence the properties of the final composite. The method we have used to purify the MWCNTs is by using the hydrochloric acid (HCl) and nitric acid (HNO<sub>3</sub>) with 1:3 by volume as it is reported in the literature [27-29].

#### 2.3 Characterization

The X-ray diffraction (XRD) patterns of the samples are recorded on a BRUKER X-ray powder diffractometer using CuK $\alpha$  (1.54060Å) radiation. The scans of the selected diffraction peaks are carried out in the step mode. The crystallite size was calculated by the help of a Scherrer's formula [30].

#### 2.4 Calculations

The lattice constant 'a' can be obtained by using the following relation:

$$a = d_{hkl}\sqrt{h^2 + k^2 + l^2}$$
(1)

where  $d_{hkl}$  is the distance between the adjacent Millar planes (h k l).  $d_{hkl}$  that can be calculated by the relation:

$$d_{hkl} = \frac{n\lambda}{2\sin\theta}$$
(2)

with n = 1 for the cubic system, XRD data can be used to calculate the X-ray density  $\rho_x$  which is given by Smit and Wijin [31].

$$\rho_x = \frac{ZM}{Na^3} \tag{3}$$

Where M is the molecular weight and N is the Avogadro's number (6.023  $\times 10^{23}$  atoms/mol), Z is the number of molecules pet unit cell (for oxide compounds having cubic spinel structure Z = 8) and 'a' is the lattice parameter in (cm). The average particle size is determined by using the Scherrer's relation

$$\mathbf{D} = \frac{0.9\,\lambda}{\beta\cos\theta} \tag{4}$$

Where D is crystallite size,  $\theta$  is the Bragg's angle,  $\lambda$  is the wavelength of the X-ray radiation, and  $\beta$  is the line width at maximum height [32].

The porosity can be calculated by the following relation

$$\mathbf{P} = \frac{\rho_x - \rho_m}{\rho_x} \tag{5}$$

Where  $\rho_x$  the density is calculated by XRD and  $\rho_m$  is the measured density. It can be calculated by the following relation.

$$\rho_m = \frac{M}{V} = \frac{M}{\pi r^2 t} \tag{6}$$

Where M is the mass, t is the thickness and r is the radius of the pellet.

The X-ray diffraction of calcinated Mn-Zn ferrite sample sintered at 1000°C are subject to calculate the average particle size using Debye – Scherrer formula. The crystalline structure of composite Nano particles is characterized by Scanning Electron Microscope (SEM).

## 3. Results and discussions

The x-ray diffraction measurement of ferrite fired at 1000°C shows that all peaks of  $Mn_{0.5}Zn_{0.5}Fe_2O_4$  consist with those of a typical spinel face centered cubic structure. Fig. 1 illustrates the XRD patterns of the ferrite, carbon nanotubes and composites having weight percent 1%, 5% and 9% of carbon nanotubes. The XRD pattern of the ferrite indicates that the material is based on cubic spinel structure. The average crystallite size is in the range of 20-35nm. The strong diffraction peaks at  $2\theta = 17.73^{\circ}$ ,  $30.22^{\circ}$ ,  $35.53^{\circ}$ ,  $43.30^{\circ}$ ,  $53.58^{\circ}$ ,  $57.35^{\circ}$ ,  $62.72^{\circ}$ ,  $72.95^{\circ}$  corresponds to (220), (311), (400), (422), (511), (440), (533), (444) typical planes of Mn-Zn ferrite spinel structure according to the standard card JCPDS file no. 520278 [33, 34].

The broadened peak at  $2\theta = 26.13^{\circ}$  indicates that the carbon nanotubes are present and no deformation takes place in their structure. The broadness of the peak indicates that the magnetite crystallites are significantly small. In Fig. 1 all the XRD patterns of the (a) composite which is formed by mixing the ferrite with 1% weight ratio of multiwall carbon nanotubes (b) composite which is formed by mixing the ferrite with 5% weight ratio of multiwall carbon nanotubes (c) composite which is formed by mixing the ferrite with 5% weight ratio of multiwall carbon nanotubes (c) composite which is formed by mixing the ferrite with 9% weight ratio of multiwall carbon nanotubes (c) composite which is formed by mixing the ferrite with 9% weight ratio of multiwall carbon nanotubes (e) ferrite (d) Multiwall Carbon Nanotubes (MWCNTs). The XRD pattern of MWCNTs (JCPGS 01-0640) purified by HCl and HNO<sub>3</sub> with volume ratio 1:3 is shown in Fig. 1(d) and a clear image is shown in the inset of Fig.1. The inter-plannar spacing corresponding to (002) plane is found to be 3.35Å like pure graphite. XRD patterns shows an overlapping of the peaks, when we have mixed only 1% of MWCNTs the (002) plane peak is not prominent but on increasing the further amount of MWCNTs the (002) plane peaks become prominent and the main peak of the spinel cubic structure reduces in height due to the decrease in grain size or decrease in crystallinity. Its mean the increasing amount of multiwall carbon nanotubes has a great influence on the structural properties of the ferrite.



Fig.1 XRD patterns of (a)  $Mn_{0.5}Zn_{0.5}Fe_2O_4$ , (b) MWCNTs, (c) Composite (Ferrite and MWCNTs 1%) (d) Composite (Ferrite and MWCNTs 5%) (e) Composite (Ferrite and MWCNTs9%), In the inset figure is MWCNTs.

The Scanning Electron Microscope (SEM) images are shown in Fig. 2. The SEM image of a pure ferrite is shown in Fig. 2(a) and the images of the composites are shown in Fig. 2(b-d) having percentage of multiwall composite 1%, 5% and 9%, respectively. The SEM image of a pure multiwall carbon nanotube is shown in Fig. 2(e). The images show that the multiwall carbon

nanotubes make agglomerates. All the obtained results are presented in Table 1. The lattice parameter, crystallite size and density decreases with the increase of multiwalled carbon nanotube concentrations. Porosity increases with the increase of MWCNTs concentrations.

 Table 1. The lattice parameter 'a', Crystallite Size D (nm), X-ray density (gm/cm<sup>3</sup>), Actual density (gm/cm<sup>3</sup>)

 and porosity values of Ferrite and composites are given below.

Sample Parameter	Ferrite	MWCNTs	Comp1%	Comp 5%	Comp 9%	
Lattice Parameter 'a' (Å)	8.0		5.0	4.8	4,2	
Crystallite Size D (nm)	30	18	28	25	22	
X-ray density $(gm/cm^3)$	5.10		3.75	2.56	2.12	
Actual density (gm/cm <sup>3</sup> )	2.85	0.02	1.92	1.06	0.43	
Porosity (%)	0.44		0.48	0.51	0.80	



Fig. 2 Scanning Electron Microscope (SEM) images of (a)  $Mn_{0.5}Zn_{0.5}Fe_2O_4$ , (b)  $Mn_{0.5}Zn_{0.5}Fe_2O_4$ , MWCNTs 1%, (c)  $Mn_{0.5}Zn_{0.5}Fe_2O_4$ , MWCNTs 5%, (d)  $Mn_{0.5}Zn_{0.5}Fe_2O_4$ , MWCNTs 9%, (e) MWCNTs.

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The Scanning electron micrograph images are shown in Fig.2. It is cleared from SEM micrographs that the samples prepared with co-precipitation technique have the spherical morphology with narrow size distribution. The particle size of the sample was in the range of 20nm to 30nm. The cylinderical structure of Multiwalled carbon nanotubes can be seen intact with the quasi spherical  $Mn_{0.5}Zn_{0.5}Fe_2O_4$  nanoparticles with small dimension have been attached on the surface of multiwalled carbon nanotubes. Agglomeration, coalescence of the particles occurred in the samples which have more percentage of carbon nanotubes.



Fig. 3 Thermo gravimetric (TG) and differential scanning calorimetric studies (DSC) (a)  $Mn_{0.5}Zn_{0.5}Fe_2O_4$ , MWCNT 1% (b)  $Mn_{0.5}Zn_{0.5}Fe_2O_4$ , MWCNT 5% (c)  $Mn_{0.5}Zn_{0.5}Fe_2O_4$ , MWCNT9%.

TG and DSC studies were made on STA 409 Cd and the heating rate was kept at 10°C/min in Ar atmosphere. Fig 3(a) shows the TG and DSC curve of the composite having 1% MWCNTs, this graph shows a weight loss of 4.184% takes place at 480°C. The ferrite composite with 5% weight of MWCNTs is shown in Fig 3(b) shows that the stability increases with the addition of more carbon nanotubes. This composite remain stable till 490°C and the weight loss is 23.51%, its mean the material is being lighter with the addition of MWCNTs. The composite with 9% weight of MWCNTs is stable till 500°C and the weight percentage loss is 22.22%. This may be due to dehydration, removal of OH ions and some organic residues. DSC curve of the samples show the exothermic peaks at 596.02°C, 611.51°C and 619.45°C. are due to the hydrocarbon residues.



Fig.5 FT-IR curves for Mn<sub>0.5</sub>Zn<sub>0.5</sub>Fe<sub>2</sub>O<sub>4</sub>

FTIR spectroscopy is a very useful technique to deduce the structural investigation and redistribution of cations between octahedral and tetrahedral sites of the spinel structure in  $Co_{0.5}Mn_{0.5}Fe_2O_4$  nanoparticles. The band around 1371cm<sup>-1</sup> is for C-H out of plane deformation vibration. The peak at 2929 cm<sup>-1</sup> is assigned to methylene (CH<sub>2</sub>) group. The adsorbed water is featured by bands at 3416cm<sup>-1</sup>, 1626cm<sup>-1</sup>which are assigned to the O-H stretching and H-O-H bonding modes of vibration, respectively. The characteristics peaks of Zn-O stretching vibration is observed at 2353 cm<sup>-1</sup>. The presence of band at 1048 cm<sup>-1</sup> represents the C-O stretching and the band at 593cm<sup>-1</sup> is attributed to the stretching vibration of tetrahedral and octahedral groups [35-38].



Fig.5 M-H curves for  $Mn_{0.5}Zn_{0.5}Fe_2O_4$  and multiwalled carbon nanotubes composites.

The specific M-H curve for ferrite multiwall carbon nanotube composite obtained from physical property measurements (PPMs) technique is shown in Fig.5. The magnetic properties are investigated at room temperature using PPMs with an applied field of -8KOe $\leq$ H $\leq$ 8KOe. The sample exhibit linear magnetization with negligible coercivety indicating that ferrite nanoparticles are super paramagnetic the magnetic domains are based on randomly oriented non-interacting particles. The samples can not fully saturate at 8KOe, this thing indicate the presence of super paramagnetic and single domain particles [35,36]. The values of saturation magnetization increased from 2.26 emu/g to 9.35 emu/g with the increased percentage quantity of multiwall carbon nanotubes. The coercivety value for the Nano composite increases from 48 Oe to 152 Oe, showing

increased in magnetization results from very well interaction between Nano crystalline  $Mn_{0.5Zn0.5}Fe_2O_4$  and multiwall carbon nanotube. The values of coercivity are so small at room temperature even can be neglected, which exhibits characteristic of super paramagnetic and their potential applications may be in electromagnetic devices, biomedical fields such as clinical diagnosis and electrochemical bio sensing.

## 4. Conclusions

Samples of Nano crystalline  $Mn_{0.5}Zn_{0.5}Fe_2O_4$  has been successfully synthesized using coprecipitation method and the effect of multiwall carbon nanotube composite have been studied. XRD pattern indicate a transition in the structure of the ferrite after the loading of the multiwalled carbon nanotubes. The saturation magnetization increases from 2.26 emu/g to 9.35 emu/g with the increase of multiwall carbon nanotubes. The thermal stability of the material increases with the increase of multiwall concentrations. Materials exhibit the super paramagnetic behavior. The coercivity value increases from 82 Oe to 152 Oe with the increase percentage of multiwalled carbon nanotubes (MWCNTs). The synthesized ferrite sample by the co-precipitation technique exhibits the single phase Face center cubic structure. The crystallite size range was 20nm to 35nm which reflect the super paramagnetic behavior as reported in the literature [38-44]. The lattice parameter, crystallite size and density decreases with the increase of multiwalled carbon nanotube concentrations while the porosity increases with the increase of MWCNTs concentrations. The increasing concentration of MWCNTS makes the material light in weight.

#### Acknowledgement

Corresponding author (Ghulam Murtaza) is thankful to Higher Education Commission (HEC) of Pakistan for providing financial assistance through IRSIP scholarship and the Zhejiang University of China for providing opportunity to do work in Carbon Black Polymer Composite Lab (CBPCL).

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