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PHYSICAL PROPERTIES CHARACTERIZATION OF $Mg_{0.9}Mn_{0.1}Fe_2O_4$ SOFT FERRITE SYSTEM

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Abstract: *Ferrite samples of the chemical composition $Mg_{0.9}Mn_{0.1}Fe_2O_4$ were prepared by standard solid state sintering technique. Wet milling for 6 h was employed to get better homogenity. The raw ferrites were sintered at 1050^{0}C for 24 h using a heating rate of 2^{o}C/min. The sintered samples were studied by means of X-ray diffraction, particle size analysis and Field Emission Scanning Electron Microscope (FESEM). Density and porosity measurements were done by Archemede's method. The microstructure were rather porous. X-Ray diffraction analysis confirmed a single phase cubic structure. Magnetic measurements were obtained by Physical Properties Measurement System (PPMS). Magnetization results exhibit collinear ferrimagnetic behavior. The dielectric constant, ε' and loss tangent, tan δ show strong frequency dependence.*

Key Words: ***solid state reaction / magnetic properties /magnesium/manganese/ferrite***

1. INTRODUCTION

In general, spinel ferrites have extensive applications in the construction of non-reciprocal devices at microwave frequencies [1]. Ferrimagnetic cation substituted spinel ferrites, such as Mg_{1-x} Mn_x Fe_2O_4, are technologically versatile materials [2]. It is known that the magnetic properties of spinel ferrites are strongly dependent on microstructure. The interesting physical and chemical properties of ferrispinels arise from their ability to distribute the cations among the available tetrahedral (A) and octahedral (B) sites [3].

Hence, an attempt has been made to investigate the synthesis, structural, magnetic and dielectric properties of the magnesium-manganese ferrite by X-ray diffraction, magnetization and dielectric constant measurements. The present work reports the investigations carried out in $Mg_{0.9}$ $Mn_{0.1}Fe_2O_4$ soft ferrite system.

2. METHODS AND MATERIALS

Samples with the general formula $Mg_{0.9}Mn_{0.1}Fe_2O_4$ were prepared by a standard ceramic technique involving a 6h- wet milling procedure. The starting materials were analytical reagent grade oxides: ferric oxide (Fe_2O_3)(Fluka), magnesium oxide (MgO)(Merck), and manganese oxide (MnO)(Aldrich). MgO (45 mole %), MnO (5 mole %) and Fe_2O_3 (50 mole %) were mixed and ground by wet milling for 6 h. This was carried out in a polyethylene bottle with zirconia balls. Distilled water was used to prepare the mixture into a slurry. The slurry prepared was dried and transferred to a porcelain crucible and pre-sintered at 900^{0}C for 12 h with a heating rate of 3°C/min. The ferrite produced is usually in lump form. It was ground into powder in a dry ball mill for 2 h. Besides reducing the particle size to $\leq 1\mu m$, grinding also eleminates intraparticle pores and homogenizes the ferrite by mixing. To promote successful sintering, the powder was well characterized after grinding with respect to particle size distribution, particle shape, homogenity, impurity and intraparticle porosity. The material was granulated through seives of 60-80 mesh BSS (250-180 μm approx.) and the granules were compressed uniaxially under a pressure of 100 MPa in a 16 ± 0.1mm diameter disc-pellet. As for ring shape, toroids of 15.2 ± 0.01mm inner diameter and 19.5 ± 0.01mm outer diameter were made. The pellets and toroids were finally sintered at 1050^{0}C for 24 hours duration. The surfaces of all the samples were ground to remove any oxide layer formed during sintering. The X-ray diffraction patterns for all the samples were recorded on a Siemen D-5000 X-ray diffractometer. The particle

size analysis was performed using a HELOS WINDOX 5. The porosity and density measurements were performed by Archemede's method. Magnetization was obtained by PPMS. The dielectric constant, ε' and loss tangent, $tan\ \delta$ were acquired by Agilent 4284A Precision LCR meter.

3. RESULTS AND DISCUSSIONS

3.1 X-ray analysis

The raw ferrite and the sintered pellets were characterized by XRD using Cu K_α radiations (λ=1.5406A°). Figure 1 illustrates the XRD patterns of $Mg_{0.9}\ Mn_{0.1}Fe_2O_4$ for 900°C and 1050°C. Analysis of X-ray diffractograms reveals the formation of single-phase cubic spinel, showing well defined reflection of allowed planes at 1050°C. But the single phase had not been formed at sintering temperature of 900°C.

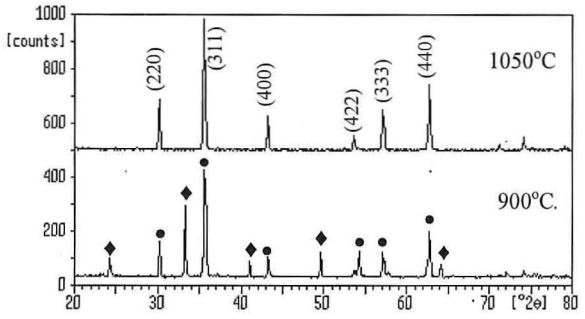


Figure. 1 *XRD patterns of $Mg_{0.9}\ Mn_{0.1}Fe_2O_4$ ($\bullet$ = $Mg_{0.9}\ Mn_{0.1}Fe_2O_4$, $\blacklozenge$ = Fe_2O_3)*

The lattice parameter was calculated from XRD data. The X-ray density d_x is also calculated according to the formula [3],

$$d_x = \frac{8M}{Na^3} \qquad (1)$$

Where M is the molecular weight, N is the Avogadro's number, 'a' is the lattice constant and '8' is the number of formula units within the unit cell of spinel structure. The values of X-ray density and the latice constant are reported in Table 1. The obtained lattice parameter value is similar to the lattice value of main system, Mg Fe_2O_4 reported by EvA-PDF file.

Table 1. *Lattice constant 'a' and X-ray density 'd_x'*

X-ray density $Mg_{0.9}Mn_{0.1}Fe_2O_4$	4.5936 g/cm^3
Lattice constant [$Mg_{0.9}\ Mn_{0.1}Fe_2O_4$]	8.3737Å
Lattice constant [[Mg Fe_2O_4]	8.392Å

3.2 Microstructure

Microstructure study is essential for optimizing the properties of final product of ferrite for various applications. The microstructure of $Mg_{0.9}\ Mn_{0.1}Fe_2O_4$ is shown in Figure 2. Both micrographs shows a typical porous structure with many intergrain pores. It is also observed that the intergranular pores are linked through the large pores. The pore structure should be regarded as interconnected voids that form a kind of capillary tubes. This structure is preferable for the adsorption and condensation of water vapour. The adsorption of water vapour can enhance the surface electrical conductivity and dielectric constant of the metal oxides. Those are some major requirements for a good humidity sensor [4].

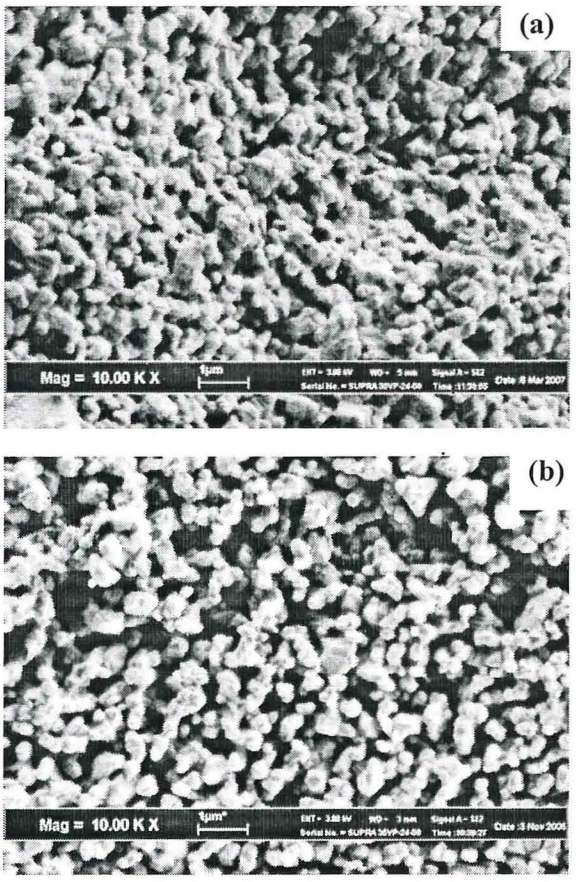


Figure. 2 *SEM micrographs of $Mg_{0.9}Mn_{0.1}Fe_2O_4$ (a) fracture surface and (b) top surface*

Table 2 represents density, porosity and water absorption measurements. The density and porosity strongly depend on sintering temperature. The relative density (i.e. Bulk density / X-ray density) is only 53.39 % which further proves the porous structure of $Mg_{0.9}\ Mn_{0.1}Fe_2O_4$.

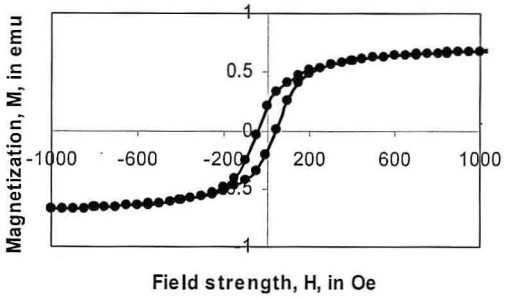
Table 2. *Density, porosity & water absorption data*

Bulk density	2.4527 g/cm^3
Apparent density	4.4236 g/cm3
Apparent porosity	44.5534 %
Water absorption	18.1704 %

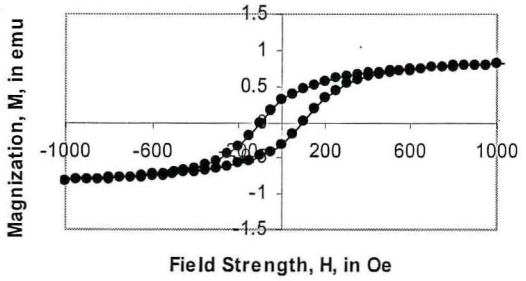
3.3 Magnetization

The behavior of a ferrimagnet in a magnetic field is considered the primary factor in the practical evaluation of the material. For this evaluation, the magnetization M was plotted against the magnetizing field H as shown in Figure 3. As the magnetic field was increased, the magnetization increases. At high fields, the magnetiza tion flattened out at the saturation magnetization, M_s.

After the material was saturated, the field was reduced to zero and and then reversed in the opposite direction to obtain the hysteresis loop. The value of coerceivity H_c and remanent magnetization M_r for three different temperatures are listed in Table 3. The characteristic of a soft material is low coercivity H_c and high permeability, resulting in a hysteresis loop which is narrow at the waist. Materials with H_c values less than $400Am^{-1}$ (5Oe) are definitely considered soft and those with H_c values greater than 8000 Am^{-1} (100Oe) are labeled hard. For materials showing coercivity values between 400 and 8000 Am^{-1} other criteria, such as the energy product B_r H_c must be considered to establish a clear classification [5]. In this case, H_c value is greater than the limit for soft ferrite and so the energy product must be considered and yet to be studied. Besides, it was observed that the increase in the value of coerceivity H_c, remanent magnetization M_r, magneton number n_B and saturation magnetization M_s with the decrease in temperature shows the importance of thermal contribution to the magnetization process.



(a)



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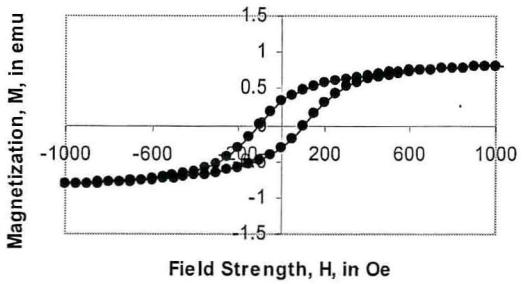


(c)

Figure. 3 *Magnetization curves of $Mg_{0.9}Mn_{0.1}Fe_2O_4$ at (a) 298K, (b) 150K and (c) 5K*

The values of magneton number n_B (saturation magnetization per formula unit in Bohr magneton) were obtained from hysteresis loop, by using the following equation [6].

$$n_B = \frac{Molecular\ weight \times saturation\ magnetization}{5585} \quad (2)$$

Table 3. *The value of satuation magnetization M_s, remanent magnetization M_r, the magneton number n_B and coerceivity H_c in different temperatures*

	298K	150K	5K
M_s (emu/g)	24.2877	30.3985	30.5508
M_r (emu)	0.2073	0.3085	0.3238
H_c (Oe)	50.0530	99.9845	100.0780
n_B (μ_B))	0.8831	1.1052	1.1108

3.4 Dielectric constant, ε' and Loss tangent, tan δ

The variation of dielectric constant ϵ_r was studied in the frequency range 1 kHz to 1 MHz at room temperature. Figure 4 shows the dielectric constant as a function of frequency. The dielectric constant initially decreases rapidly with increase in frequency but remains fairly constant beyond 10 kHz. It is evident from these results that a dielectric dispersion was observed at low frequency region. This behavior of frequency dependence of ε' quite agrees with the well known frequency dependence of spinel ferrite [7].

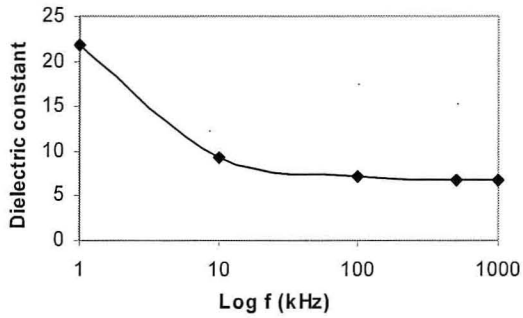
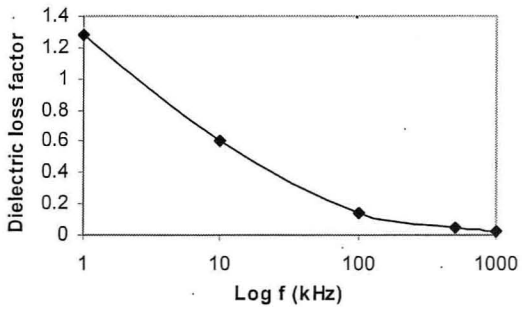


Figure 4 *Variation of dielectric constant, ε' with the frequency*



Figuure. 5 *Dielecrtic loss factor, tan δ as a Functtion of frequency*

Figure 5 depicted the variation of dielectric loss tangent tan δ against log frequency at room temperature. The dielectric loss decreases with increasing frequency. This is also an evident for a normal dielectric behavior with frequency [8].

4. CONCLUSION

(1) Wet milling for 6 h can provide single phase cubic spinel ferrite at final sintering 1050^0C for 24 hours with 2^0C /min.
(2) The structure formed at 1050^0C is porous.
(3) Magnetization measurements exhibit Neel's ferri -magnetic structure.
(4) The dielectric constant (ε') and loss tangent *tan δ* show strong frequency dependence.
(5) The microstructure and the material properties show the applicability as humidity sensing material.

5. ACKNOWLEDGEMENTS

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