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Comparative Study Of The Structural And Magnetic Properties Of Magnesium Ferrite Prepared By Ceramic And Sol-Gel Auto Combustion Technique

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ABSTRACT

Magnesium ferrite ($MgFe_2O_4$) in bulk and nanocrystalline form has been synthesized by standard ceramic and sol-gel auto combustion method respectively. X-ray diffraction (XRD), scanning electron microscopy (SEM) and pulse field hysteresis loop technique were employed to investigate structural and magnetic properties. The X-Ray diffraction pattern of bulk and nano size Mg ferrite indicates that the bulk sample shows sharp and intense Bragg's reflection whereas nano size samples show slightly broader Bragg's reflection. The analysis of X-ray diffraction pattern revealed the formation of single phase cubic spinel structure for both the samples. The particle size evaluated using Scherrer's formula confirms the bulk and nanocrystalline nature of the prepared magnesium ferrite. The scanning electron microscopy technique proves the nanocrystalline nature of the sol-gel prepared magnesium ferrite. The bulk magnesium ferrite requires high sintering temperature of the order of 1050 °C whereas nano size Mg ferrite is sintered at sufficiently low temperature i.e. 600 °C. The sintering temperature affects the physical properties of magnesium ferrite as evidenced from the X-ray density, bulk density and porosity values. A comparative study of the structural and magnetic properties of bulk and nano size magnesium ferrite is presented in this work.

Keywords: Magnesium ferrite, Nanocrystalline, Ceramic technique, Sol-gel auto combustion technique, Comparative study.

INTRODUCTION

Spinel ferrites with general chemical formula MFe_2O_4 have attracted scientist and technologist because of their attractive, interesting and useful combined electrical as well as magnetic properties. They possess high electrical resistivity, low eddy current and dielectric losses, high saturation magnetization, high permeability and high Curie temperature etc properties. These properties of spinel ferrites are sensitive to many parameters. The most influencing parameter is the method of preparation [1]. Spinel ferrites can be synthesized by number of synthesis techniques. The most common method of synthesis for spinel ferrite is ceramic method. The ceramic method has some inherent drawbacks like high sintering temperature, in-homogeneity, time consuming, etc. These drawbacks of the ceramic method can be eliminated or minimized in wet chemical methods like sol-gel auto combustion technique. The sol-gel auto combustion method requires less time and low temperature for synthesis, produces homogenous and nano size samples, the method is low cost and chemically stable. In the recent years nanoparticles of spinel ferrite have been synthesized on large scale for different applications using wet chemical methods [2-5]. The nanoparticles of spinel ferrites have applications in several fields. They can be used in magnetic drug delivery, sensors, catalyst, computer memory chips [6-7]. Extensive work has been carried on synthesis, characterization and magnetic properties of magnesium ferrite [8-9]. Magnesium ferrite ($MgFe_2O_4$) is a unique spinel ferrite with random distribution of cations over the available tetrahedral (A) and octahedral [B] sites. The degree of inversion depends on synthesis methods and sintering temperature. The properties of magnesium ferrite prepared by ceramic method and sol-gel auto combustion method are different from each other because of their particle size order. To understand the effect of particle size a comparative study of the structural and magnetic properties of Mg ferrite prepared by ceramic technique and sol-gel auto combustion method. In the literature comparative study of magnesium ferrite is reported [10].

MATERIALS AND METHODS

The samples of magnesium ferrite in bulk and nano size form have been prepared by ceramic method and sol-gel auto combustion method using high purity of oxides and nitrates respectively.

Ceramic method: The sample of $MgFe_2O_4$ spinel ferrite was prepared by ceramic method (double sintering method) using AR grade oxides of respective ions i.e. MgO and Fe_2O_3. These oxides were accurately weighed on an electronic balance and mixed together according to stoichiometric proportions. The mixed oxides were finely ground for 3-4 h using Agate mortar and pestle. The ground powder is then pre-sintered at 950 ^{0}C for 24 h in a programmable furnace and cooled slowly to room temperature. The sintered powder is again reground for 3-4 h and finally sintered at 1050 ^{0}C for 24 h followed by slow cooling. The sintered powder is pressed into cylindrical form using hydraulic press and by applying a pressure 6 ton. Finally sintered powder and pellets were used for characterization and for the measurement of structural and magnetic properties.

Sol-gel auto combustion method: In case of sol-gel auto combustion method AR grade nitrates of respective ions were taken. The citric acid was used as fuel for the auto combustion reaction. The nitrates were accurately weighed on an electronic balance and dissolved together according to stoichiometric proportions in minimum amount of water. The mixture of the nitrates was then stirred continuously and the citric acid solution was added gradually to it so as to neutralize the precursor solution by adjusting its pH value to 7. The sol thus formed was also heated to a temperature of 80 ^{0}C while it is being stirred continuously. Due to heating of the sol its water content gets evaporated and the sol gets converted in to viscous gel. The heating is continued till the ignition of auto combustion reaction. Once the auto combustion reaction gets ignited the gel burns out within few minutes leaving the loose voluminous ferrite powder behind as the end product of the sol-gel auto combustion reaction. The as-prepared ferrite powder is then ground for about 1 h and sintered at 600 ^{0}C for 12 h in a programmable furnace and then cooled slowly to room temperature. The sintered powder is pressed into cylindrical form using hydraulic press and by applying a pressure 6 ton.

RESULTS AND DISCUSSION

The single phase cubic spinel structure of the prepared Mg ferrite was confirmed by X-Ray diffraction method. The X-Ray diffraction patterns of bulk fig. 1 (a) and nano size fig. 1 (b) Mg ferrite indicates that the bulk sample show sharp and intense Bragg’s reflection whereas nano size samples show slightly broader Bragg’s reflection.

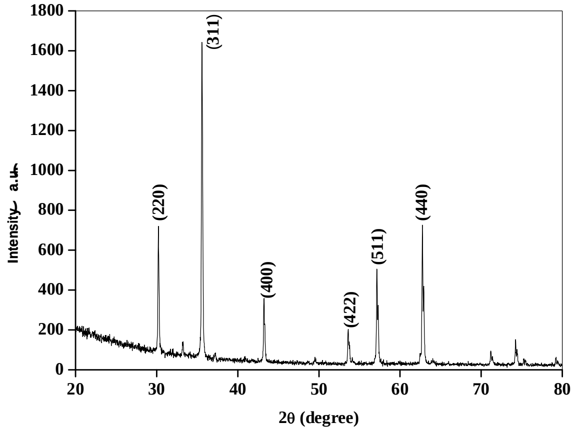

Fig 1 (a). X-ray diffraction pattern of bulk size $MgFe_2O_4$ spinel ferrite system

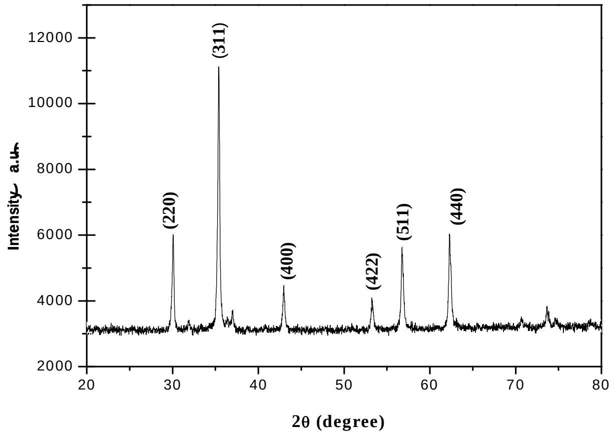

Fig.1 (b). X-ray diffraction pattern of nano size $MgFe_2O_4$ spinel ferrite system

The difference in XRD patterns may be due to different method of preparation. Using XRD data the structural parameters were evaluated and few of them like lattice constant, particle size, X-ray density, bulk density and porosity have been compared. Table 1 illustrates comparative data of the selected structural parameters for bulk and nano size Mg ferrite.

Table 1. Comparative data of the structural parameters of bulk and nano size $MgFe_2O_4$ spinel ferrite.

Structural Parameters	**$MgFe_2O_4$**	
	Bulk	**Nano**
Lattice constant ‘a’ (Å)	8.366	8.335
X-ray density ‘d_X’ (gm/cm^3)	4.539	4.553
Bulk density ‘d_B’ (gm/cm^3)	3.275	2.832
Porosity ‘P’ (%)	27.77	37.8
Particle size ‘t’ (nm)	409	38

It is evident from this table that the lattice constant of the bulk Mg ferrite is slightly higher than that of nano size Mg ferrite. Similarly, X-ray density of the bulk Mg ferrite is found to be less than that of nano-size Mg ferrite. The bulk density of the bulk Mg ferrite is greater than that of nano size Mg ferrite. The porosity of the bulk Mg ferrite is also less than that of nano size Mg ferrite.

The particle size of the bulk size Mg ferrite is of the order of 400 Å whereas it is 38 nm for nano size Mg ferrite. The observed variation in the structural parameters investigated for bulk and nano size Mg ferrite can be attributed to the varying different particle size arising due to different method of preparation. The bulk Mg ferrite requires high sintering temperature of the order of 1050 °C whereas nano size Mg ferrite is sintered at sufficiently low temperature i.e. 600 °C. The difference in sintering temperature can cause variation in bulk density and thereby change in porosity.

The magnetic properties like saturation magnetization, remanence magnetization, coercivity were obtained using pulse field hysteresis technique at room temperature. The magnetizations versus applied magnetic field (M-H) plots for bulk and nano size spinel ferrite system are shown in fig. 2 (a) and fig. 2 (b) respectively.

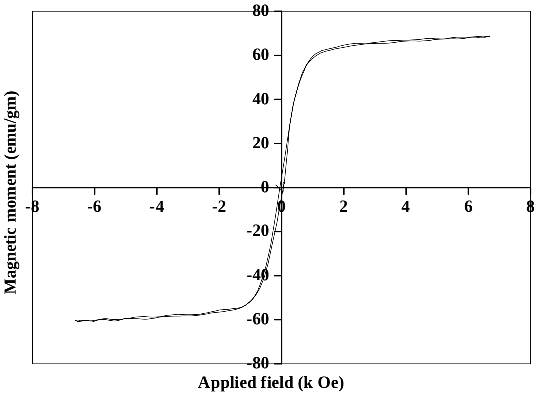

Fig 2 (a). Hysteresis loop of bulk size $MgFe_2O_4$ spinel ferrite system**.**

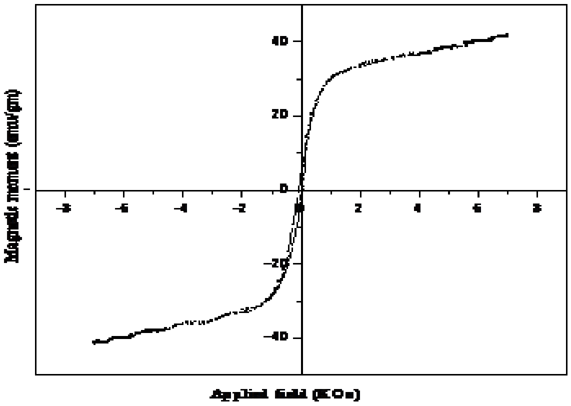

Fig 2 (b). Hysteresis loop of nano size $MgFe_2O_4$ ferrite system

Table 2 represents the magnetic parameters like saturation magnetization (Ms), coercivity (Hc), remnant magnetization (Mr), etc obtained from M-H hysteresis plots recorded at room temperature.

Table 2. Comparative data of the magnetic parameters of bulk and nano $MgFe_2O_4$ spinel ferrite system.

Magnetic Parameters	**$MgFe_2O_4$**	
	Bulk	**Nano**
Saturation magnetization 'Ms'(emu/gm)	68.71	41.66
Remanent magnetization 'Mr'(emu/gm)	2.19	1.58
Mr/Ms	0.031	0.038
Coercivity 'Hc'(Oe)	77.33	132
Magneton number 'n_B'(μ_B)	2.46	1.91

It is evident from table 2 that the saturation magnetization (Ms) of bulk size Mg ferrite is of the order of 68 emu/gm whereas, for nano size Mg ferrite it is of the order of 41 emu/gm. The coercivity (Hc) of the bulk Mg ferrite is of the order of 77.33 emu/gm while it is of the order of 132.96 emu/gm for nano size Mg ferrite. The magneton number (n_B) calculated using saturation magnetization and molecular weight also shows the difference in their values. The large value of coercivity in case of nano size Mg ferrite is attributed to the nanocrystalline nature. The observed low value of saturation magnetization for nano size Mg ferrite can be attributed to the distribution of Mg^{2+}, Fe^{3+} and there percentage over the tetrahedral (A) and octahedral (B) sites. Thus, it is observed from the experimental data on structural, electrical and magnetic properties of bulk and nano size Mg ferrite that the method of preparation strongly influences the properties of Mg ferrite which produces particles of different size. The sintering temperature also affects the properties of Mg ferrite. The change in the structural and magnetic properties of Mg ferrite may be attributed to the method of preparation, sintering temperature and particle size effect. Our results are in analogy with the size induced effect on nanocrystalline cobalt ferrite [11].

APPLICATIONS

Magnesium ferrite ($MgFe_2O_4$) prepared by sol-gel method in nanocrystalline form possess good structural and magnetic properties. The crystallite size, saturation magnetization, coercivity and remanence magnetization of the prepared sample can have applications in the field of magnetic recording media, inductors and transformer cores.

CONCLUSIONS

Magnesium ferrite ($MgFe_2O_4$) was successfully prepared in bulk and nano size by using ceramic and sol-gel auto combustion method. The comparative study of the structural and magnetic properties of bulk and nano size $MgFe_2O_4$ prepared by ceramic technique and sol-gel auto combustion technique showed the difference in various structural and magnetic parameters. The structural and magnetic properties of the nano size Mg ferrite look to be superior and advantageous as compared to bulk size Mg ferrite.

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