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SUPER PARAMAGNETISM BEHAVIOUR IN MICROWAVE PROCESSED $Mn_{0.5}Zn_{0.5}Fe_2O_4$ NANOCRYSTALLINE SPINEL FERRITES

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ABSTRACT

Nanoparticles of spinel cubic $Mn_{0.5}Zn_{0.5}Fe_2O_4$ are first prepared by co-precipitation method, then subjected to conventional solid state sintering and microwave processing techniques. Particle sizes are estimated (by FESEM) to be in nanometer (6-13nm) range. The magnetization measurement by vibration sample magnetometry of the conventionally sintered sample shows paramagnetic nature at room temperature, super paramagnetic behavior at low temperature, and ferromagnetic nature at below blocking temperature. The blocking temperature is estimated to be nearly 110 K. Microwave processed samples show super paramagnetic nature even at room temperature.

Keywords: Ferrites, Microwave Processing, Nanoparticles, Superparamagnetism Spinel ferrites. PACS: 75.50.Gg, 75.60.Ej, 78.67.Bf

I. INTRODUCTION

The microwave processing is a green and economic technique wherein heat is produced homogenously in the core of a material, when it is irradiated with microwaves of suitable energy. Consequently it is a rapid and economic technique of thermally processing materials. It is capable of forming new phases through rapid decrystallization, nanostructural phase formation, mineral processing, alloy formation and inducing ferromagnetism through doping of semiconductors in a very short time (2-5 minutes) [1]-[2]. Recent interest in microwave processing of materials is highlighted by several national and international symposia that have focused on only microwave processing of materials.

Mn-Zn doped ferrite ($Mn_{1-x}Zn_xFe_2O_4$) nanoparticles are used in a wide range of industrial and biomedical applications [3]. There are several methods for preparing ferrite nanoparticles including, sol-gel method [4], hydrothermal [5]-[6], co-precipitation method [7] and ball milling method. Among these methods co-precipitation method has attracted maximum attention for preparing ferrite nanoparticles. Mn - Zn nanoparticles have been reported to be super paramagnetic [8].

The aim of the present paper is to employ the versatile microwave processing technique to modify micro-structure and hence the magnetic properties of ($Mn_{1-x}Zn_xFe_2O_4$) nanoparticles. For comparison the samples are prepared by conventional solid state sintering method also.

II. EXPERIMENTAL TECHNIQUES

The precursor of the desired nanoferrite ($Mn_{1-x}Zn_xFe_2O_4$) was prepared by chemical co-precipitation method. In this method, aqueous solutions of $Mn(CH3COO)_2.6H_20$, $Zn(NO_3)_{2.}6H_2O$ & $Fe(NO_3)_{2.}9H_20$ were prepared in de-ionized water by dissolving stoichiometric amounts of these compounds. Aqueous solution of sodium hydroxide (NaOH) was added to this solution to maintain a suitable pH and hence to obtain a hydroxide precipitate of the desired compound. A part of the solution is filtered, washed, dried and kept at 90°C for 24 hrs thus resulting in $Mn_{0.5}Zn_{0.5}Fe_2O_4$ nanoparticles. This sample is named as M0. Other parts of the solution were subjected to microwave heating (2.45GHz) in a multimode microwave oven for 10 and 15 min each. The resulting compounds were filtered, washed and dried. Microwave processed $Mn_{0.5}Zn_{0.5}Fe_2O_4$ samples were named as M1 and M2. In another process the remaining part of the main solution was filtered, washed and dried and kept for solid state sintering at 400°C for 4h and it was named as S1.

Phase formation of all samples was checked by powder x-ray diffraction (XRD) method using Cu kα radiation in the 2θ scan range from 20 to 80º (PW 1710 Philips Holland). Micro-structure/particle size has been investigated by high resolution field emission scanning electron microscopy (FE-SEM) (Model:Carl Zeiss SMT Ltd. SUPRATM 40). Low field M-H and M-T measurements were done to magnetically characterize the samples using vibration sample magnetometer with a sensitivity of 10-3 emu (SR 830 Lock in amplifier and Lakeshore 325 PID temperature controller).

III. RESULTS AND DISCUSSIONS

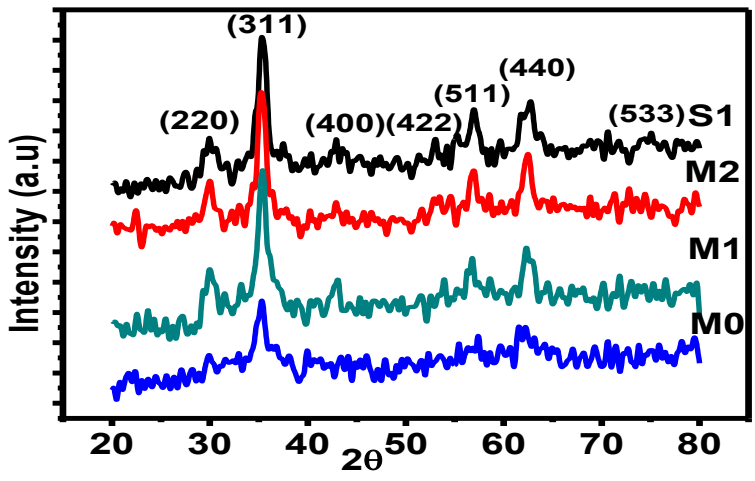


Figure1. The XRD pattern obtained for the samples

Fig. 1 shows the XRD patterns of M0, M1, M2 and S1 samples. All the peaks in these diffractograms correspond to pure cubic spinel phase of Mn0.5Zn0.5Fe2O4. Reitveld analysis reveals that no secondary phase is present in any of the samples. Considerably broadened lines in the XRD pattern are indicative of the presence of nano-size particles. The maximum intensity peak (311) of all the samples were fitted with a Gaussian shape to determine the exact peak position as well as the full width half the maximum (FWHM). The average particle sizes were estimated with the help of the Debye-Scherrer [4] equation

$$t = \frac{0.9\lambda}{B\cos\theta_B} \quad ; \qquad B = \left(B_M^2 - B_S^2\right)^{1/2}$$

where t is the thickness (diameter) of the particle, λ is the X-ray wavelength (1.54 Å), BM and BS are the measured peak broadening and instrumental broadening in radian respectively and θB in the Bragg angle of the reflection. The average grain size was 7, 9, 10 nm for M0, M1, M2 samples respectively and 10 nm for S1 sample.

Field emission scanning electron microscopy (FE-SEM) is a visual demonstration technique. It was revealed that the particle size of the four samples, such as M0, M1, M2 and S1. All samples were in nanometer range particles, particle sizes were found between 6 -13 nm range for all samples. From Fig. 2 it has been observe the particle size was found as 8nm for M0 sample. For conventionally heated sample (S1) particle size was found as 12 nm. For microwave processed nanoparticles were found to be 10 and 12 nm for M1 and M2. Particle size increases with microwave heating time.

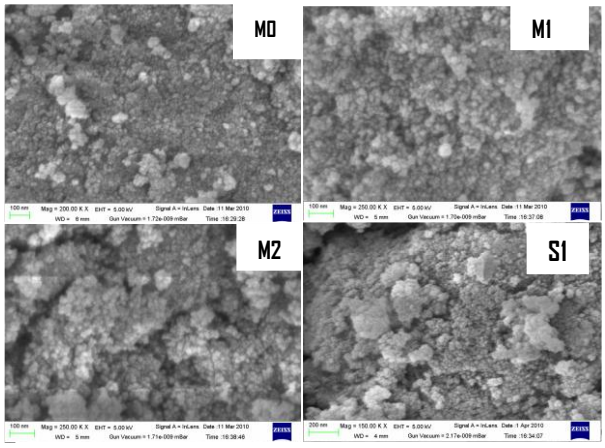


Figure2. FESEM images of the Mn0.5Zn0.5Fe2O4 samples for microwave processed samples M1, M2, M3, and solid state sintered sample S1.

Fig. 3shows a M-H graph of the M0 sample, it shows ferromagnetism nature with tiny magnetic moment, with coercivity of 35Oe (inset fig(3)) and remanence of 0.08 emu/g.

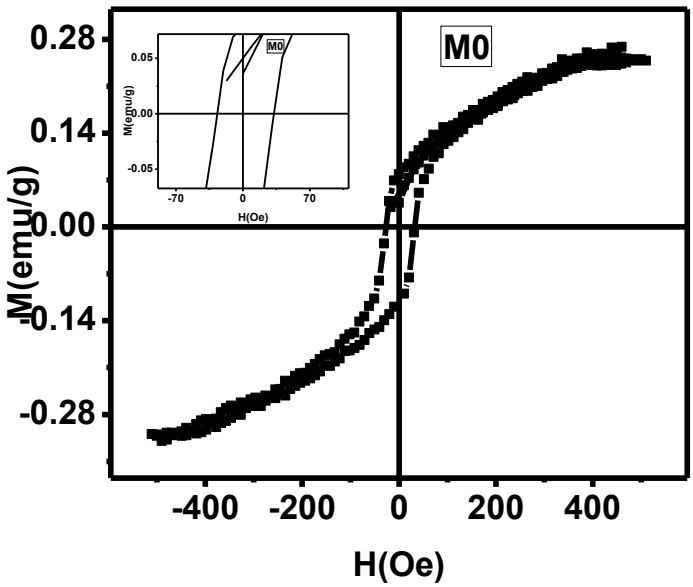


Figure3. M-H Curve of the M0 sample at room temperature.

The M-H loops for microwave processed samples (M1 and M2) are shown in Figure 4. Both the loops reveal super-paramagnetic nature of these samples. It has been observed that magnetization M at higher fields (say 500 Oe) increases when the microwave processing time is increased. It may be attributed to decrease in defects and strain in the nanoparticles with the increase in processing time and consequently to the increase in the exchange interaction between the magnetic ions. Reduced overall magnetostriction energy and anisotropy constant, are known to foster higher magnetization.

Microwave processing accelerates the formation kinetics of reaction in ceramics [9]. It may be proposed that during microwave processing the multi-domains are converted into single domain particles. We observed that the magnetic moment strongly depend upon microwave heating time. The magnetic properties are improved by microwave processing.

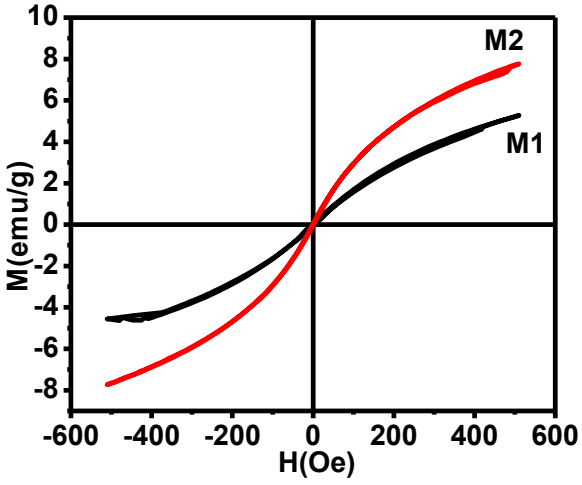
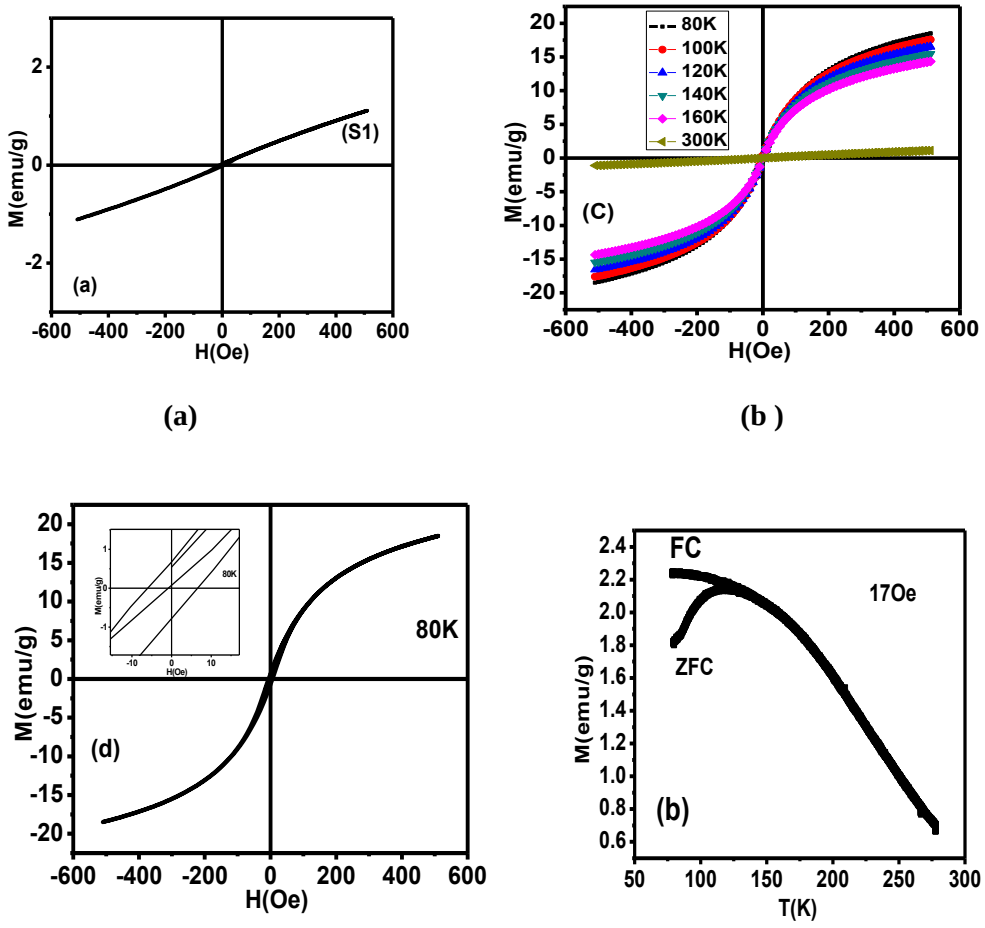


Figure4. M-H Curve of the MW processed sample M1 and M2 at room temperature.



(a) **(b)**

(c) **(d)**

Figure 5.(a) M-H curve of the sample S1at room temperature, (b) Isothermal M-H curves at different temperatures exhibiting paramagnetic (at 300 K) and super paramagnetic down to 120 K, (c) Isothermal M-H curves at 80 K exhibiting ferromagnetism (inset in the fig shows coercivity of the sample.), and (d) Thermo magnetic (M-T) curve recorded at 17 Oe and at 80K revealing blocking temperature of ~110 K.

Figure 5(a) reveals that isothermal M-H curve of the solid state sintered sample (S1) recorded at room temperature, however, exhibits paramagnetic nature. This sample exhibits super-paramagnetic at a low temperature. Figure 5(b) gives isothermal M-H curves at different temperatures exhibiting paramagnetic (at 300 K) and super-paramagnetic down to 120 K. Further lowering the temperature transforms the magnetic behavior to ferromagnetism, as depicted by an isothermal M-H curves recorded at 80 K, with a small value of coercivity. The inset in the fig shows the coercivity of the sample to be ~10 Oe.). That the ferromagnetism is exhibited below certain temperature (blocking temperature) is established by recording the field cooled and zero-field cooled (FC-ZFC) magnetization vs. temperature curves. The M-T curves in ZFC-FC modes recorded at a low field of 17 Oe, show bifurcation near 110K (fig. 5 (d))[10-12]. This is the blocking temperature at which the transformation from ferromagnetic to super-paramagnetic nature occurs. When temperature of the sample increases, the saturation magnetization decreases and it goes to paramagnetic state, because of the thermal

effects allow flips of magnetic moments between the easy magnetization directions by getting over the energy barriers in zero field. So the corresponding easy magnetization directions are randomly oriented, and total magnetization is naturally reduced by increasing the sample temperature.

IV. CONCLUSION

Mn-Zn doped ferrite (Mn1-xZnxFe2O4) nanoparticles are successfully synthesized by both conventional solid state sintering and microwave processing techniques. Microwave processing, owing to wave and matter interaction at ionic level has resulted in defect and strain free nanoparticles with improved magnetic properties. Microwave processed sample shows super paramagnetic nature at room temperature. Conventionally sintered sample shows paramagnetic nature at room temperature, super paramagnetic nature at lower temperature and ferromagnetism below blocking temperature (110 K).

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