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Grain size effects on the magnetic properties of $Zn_xMn_{1-x}Fe_2O_4$ nanoferrites

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ABSTRACT

Single phase nanoferrites bearing the chemical formula $Zn_xMn_{1-x}Fe_2O_4$ ($0 \le x \le 1.0$) have been produced under low reaction temperature of 200 °C. The compounds were characterized by X-ray diffraction, Mössbauer and SQUID measurements. The particle size varies between 5 nm and 14 nm. The particle size dependence of the magnetic properties is investigated. Our results indicate spin-glass behavior at temperatures between 100 K and 200 K. The variation of Zn concentration has also significant effects on the structural and magnetic properties. The complex variation of lattice parameter with x is explained on the basis of redistribution of Zn atoms in both tetrahedral (A) and octahedral (B) sites. The ^{57}Fe Mössbauer effect spectra show paramagnetic spin state in all the compounds.

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1. Introduction

Magnetic nanoparticles have broad practical applications in important technologies such as ferrofluids, magnetic drug delivery and high density information storage [1]. The unusual properties of nanosized compared to bulk samples have attracted a lot of attention [1–8]. The important features of magnetic nanoparticles are superparamagnetism and surface spins which can lead to canted-spin structures. In the superparamagnetic state there are weak inter-particle magnetic interactions. Super-spin-glass behavior can be observed when there is sufficiently strong magnetic interaction and the ensemble of nanoparticles shows a collective behavior [9]. Zn–Mn ferrites are suitable candidates for applications in magnetic fluids, heat transfer systems, magnetoelectric composites, etc. [2]. In this work we have produced $Zn_xMn_{1-x}Fe_2O_4$ ($0 \le x \le 1.0$) nanoferrites under low reaction temperature of 200 °C and investigated the effects of annealing conditions on the magnetic properties.

2. Experimental details

$Zn_xMn_{1-x}Fe_2O_4$ ($0 \le x \le 1.0$) nanoparticles were produced by using a hydrothermal process. The starting materials were $ZnCl_2$ (99.999%), $MnCl_2 \cdot 4H_2O$ (99.99%) and $FeCl_3 \cdot 6H_2O$ (99%). Required proportions of chlorides were mixed thoroughly to produce 1 g samples. A concentration of 5 M solution of NaOH was then slowly added to the chloride mixture solutions under rapid stirring until pH was 10. The precipitate was washed several times by deionized water and finally by 200 ml of ethanol. The clean precipitate was dispersed in 300 ml of ethylene glycol under rapid stirring. The mixture was then placed in a 600 ml stainless steel pressure vessel (Watlow series model PARR 4842 reactor). The pressure vessel was heated to 200 °C and the gage pressure was allowed to gradually rise to 100 psi. These conditions were held for 6 h. The cooled products after boiling were filtered and washed by deionized water and ethanol. The recovered synthesized powders were dried under 250 W infrared light and homogenized using an agate mortar and a pestle.

The XRD patterns of the samples were obtained using a monochromatic beam of Co-Kα radiation ($\lambda = 1.7903$ Å) on a Phillips diffractometer (type: PW1710). The XRD data were used to confirm formation of cubic spinel phase of the compounds and estimate crystal size and lattice constants. The Mössbauer spectra were recorded at room temperature (300 K) using a conventional constant acceleration Mössbauer spectrometer with a ^{57}Co source sealed in Rh matrix. The magnetization measurements were performed by using a Quantum Design SQUID magnetometer from 2 K to 380 K.

3. Results and discussion

Fig. 1 shows the variation of XRD spectra for $Zn_xMn_{1-x}Fe_2O_4$ as a function of Zn concentration (x). All the XRD peaks were successfully indexed to pure cubic spinel structure. The broad

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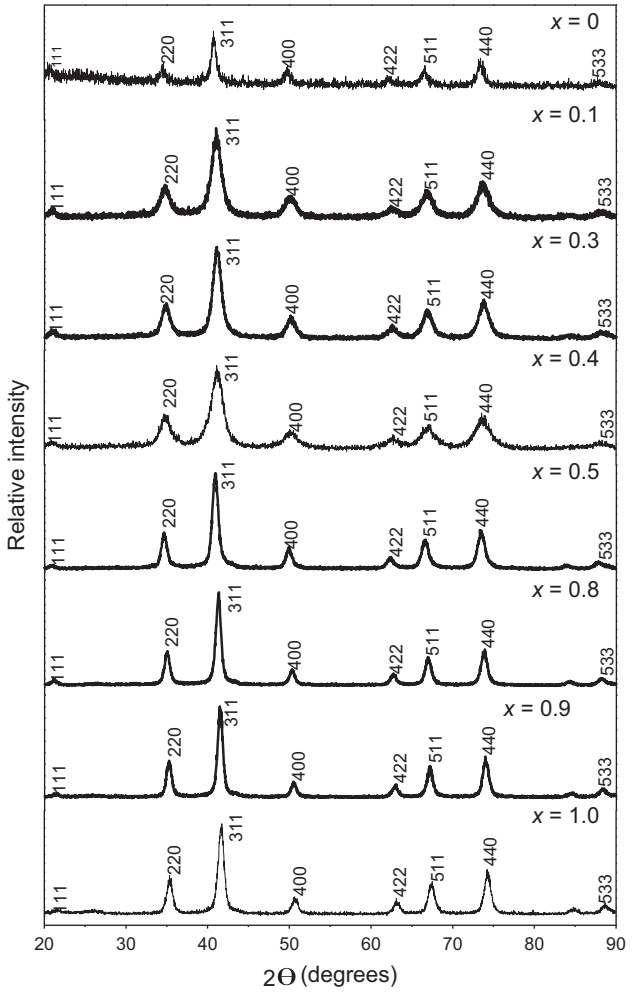

Fig. 1. XRD patterns for $Zn_xMn_{1-x}Fe_2O_4$ with x.

Table 1
Grain size (D), lattice parameter (a) and X-ray densities (ρ_X) for $Zn_xMn_{1-x}Fe_2O_4$.

x	D (nm) ± 0.02	a (Å) ± 0.01	ρ_X (g/cm^3) ± 001
0	13.81	8.53	4.94
0.1	8.20	8.47	5.07
0.3	7.65	8.45	5.15
0.4	5.42	8.45	5.17
0.5	11.99	8.49	5.11
0.6	11.26	8.49	5.13
0.7	14.08	8.47	5.20
0.8	13.48	8.42	5.35
0.9	13.48	8.38	5.42
1.0	8.59	8.35	5.50

peaks are associated with fine particles. The values of grain sizes calculated by using Debye–Scherrer equation [1] for the as-prepared system of $Zn_xMn_{1-x}Fe_2O_4$ compounds are listed in Table 1. The particle size varies between 5 nm and 14 nm. The smallest particle size of 5.4 nm was obtained for the $Zn_{0.4}Mn_{0.6}Fe_2O_4$ ($x=0.4$) compound.

The variation of the lattice parameter with x is shown in Fig. 2. A complex variation of lattice constant with increasing x is observed. The lattice constant reduces to a minimum at about $x=0.3$ and attains maximum at $x=0.6$. A decrease in lattice parameter with further substitution of Zn atoms occurs. The

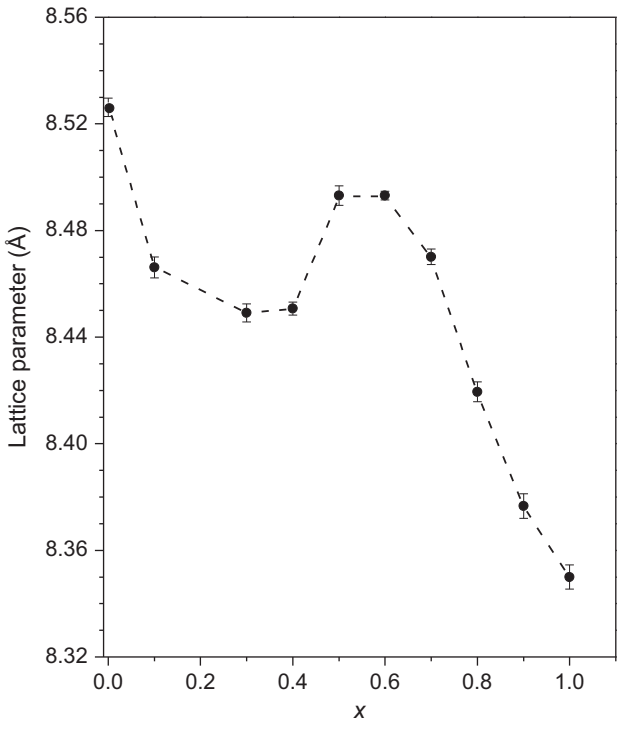

Fig. 2. Lattice parameter for $Zn_xMn_{1-x}Fe_2O_4$ oxides as function of x.

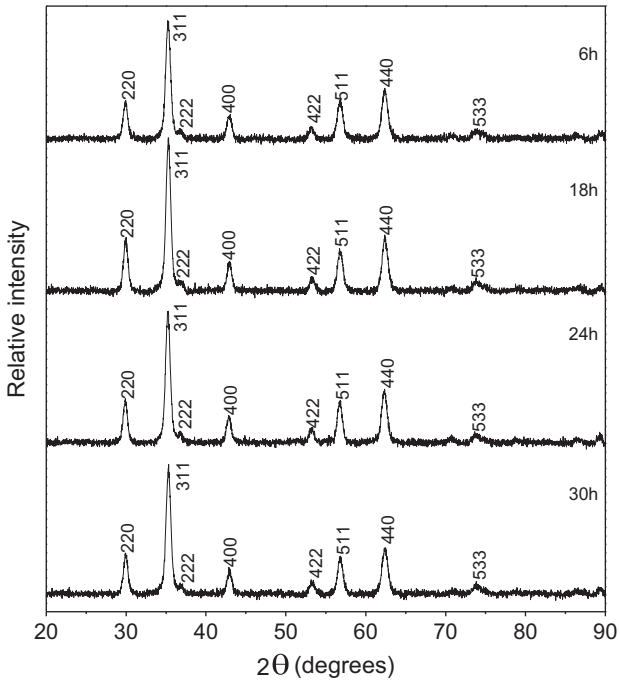

Fig. 3. XRD spectra for $Zn_{0.4}Mn_{0.6}Fe_2O_4$ as a function of annealing time.

reduction of the unit cell parameter with increasing Zn content can be explained by substitution of bigger Mn^{2+} ions (0.91 Å) by smaller Zn^{2+} (0.74 Å) ions. Similar behavior was observed in $Zn_xMn_{1-x}Fe_2O_4$ compounds produced by high energy ball milling [6]. The increasing lattice constant with increasing Zn concentration between $x=0.4$ and $x=0.6$ observed in our samples may be due to replacement of smaller Fe^{3+} (0.67 Å) by larger size Zn^{2+} (0.74 Å) at the octahedral (B) sites or partial migration of Zn^{2+} ions

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