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Synthesis of $Mn_{0.2}Zn_{0.8}Fe_2O_4$ particles by high energy ball milling and their applications

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Ultra fine $Mn_{0.2}Zn_{0.8}Fe_2O_4$ magnetic particles are developed by high energy ball milling technique and investigated for physical properties. The crystalline phase, crystallite size, surface morphology, metal oxide bonding and porosity of these magnetic particles are analyzed. The porosity increases on increasing the grinding period due to decrease in the particle size and crystallinity reduces. The IR spectra measured in the range of 4000-400 cm^{-1} exhibit symmetric stretching mode of (FeO_4) and (ZnO_4) tetrahedral at 669.7 cm^{-1} and 545.6 cm^{-1}. The lattice strain induced by ball milling process has been determined. The porosity plays an important role in chemisorption and physisorption of species on the sites of the particles. This property of these particles has been exploited for the applications of humidity sensor.

Keywords: Ferrite, Crystallite size, Ball milling, Humidity sensing, Mn-Zn ferrite, FTIR spectra

Magnetic nanoparticles are gaining importance because of their wide range of applications in high-density magnetic recording, magnetic fluids, medicine, sensors and actuators[1-3] Recently, they are often used in temperature sensitive ferrofluids for applications in heat transfer enhancement and energy conversion devices[4,5]. The nanomagnetic particles are prepared by various chemical methods, viz., as chemical co-precipitation method[6,7], sol–gel synthesis[8], citrate precursor[9], hydrothermal precipitation[10] and other chemical methods[11]. The Mn-Zn ferrites possessing a cubic spinel structure are described as $(A)[B]_2O_4$, where (A) and [B] are referred the tetrahedral and octahedral cation sites in a FCC anion (oxygen) sub-lattice.

The spinel ferrites are chemically stable, have porous structure[12] and resistive type. This type of property of these fine nanomagnetic particles can be used in humidity sensor applications. Keeping this in mind an attempt has been made to develop Mn-Zn ferrites ultrafine magnetic particles by high energy ball milling using appropriate ratio of precursor oxides. The milling time was varied and the samples were examined for the crystalline phase, crystallite size and lattice strain of these particles by XRD, FTIR and SEM spectroscopic techniques. Usually the spinel ferrites are chemically stable and are being used as resistive type humidity sensor. The electrical conduction mechanism in such sensors is described as water molecules which physisabsorb at the site of the surface over the layered chemisorbed water layer[13]. The conduction depends upon the structural, shape size and the porosity of the material. The results of our investigations on induced lattice strain due to ball milling and large ionic radii of Zn^{2+} ion on tetrahedral interstitial site are explained on the basis of XRD peak shift. The electrical conductivity of the nanomagnetic particles has been studied and correlated with structure, particles shape, size and porosity of the particles. The electrical conduction mechanism is described in terms of the adsorption (phyisorption) of water molecules at the site of surface over the chemisorbed water layer. The results of electrical resistance and porosity are correlated with relative humidity.

Experimental Procedure

Ultra fine particles of $Mn_{0.2}Zn_{0.8}Fe_2O_4$ are synthesized by grinding the metal oxides on a high energy ball mill (HEBM) with varying the milling times. A high purity chemicals of MnO, ZnO and Fe_2O_3 oxides as given in Table 1 in stochiometric proportions are taken and then mixed thoroughly using pestle and mortal before placing them for grinding in the tungsten carbide (WC) jars.

The powders were mixed with acetone to ensure the wet grinding. The weight to volume ratio of WC balls and materials is optimized as 10:1 for the

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Table 1—Weight ratio of different oxides used for the development of $Mn_{0.2}Zn_{0.8}Fe_2O_4$ as per the stoichiometric requirements

S. No.	Compound name	Calculated weight (g)			Actual weight taken (g)		
		Fe_2O_3	MnO	ZnO	Fe_2O_3	MnO	ZnO
1	$Mn_{0.2}Zn_{0.8}Fe_2O_4$	20.455	2.204	8.254	20.495	2.207	8.255

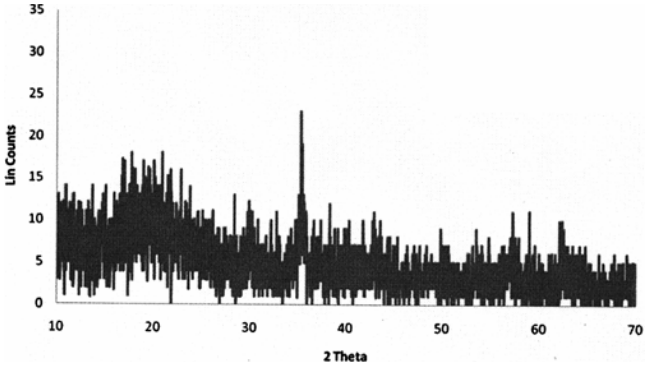

Fig. 1a–X-Ray diffraction patterns of the samples grinded for 20 h without annealing

effective grinding in Retsch Co. Planatry ball mill at 400 rpm. Samples are analyzed after 10 h (sample 'a'), 15 h (sample 'b') and 20 h (sample 'c') of grinding for the crystalline phase formation by D-8 Advanced Bruker make powder X-ray diffractmeter (XRD) using Cu-K_α radiations at 40 kV/40 mA. These powder samples are annealed at 773 K for two hours using muffle furnace (Carbolyte tubular furnace, UK) to improve the crystallinity. In order to calculate the crystallite size a slow scan rate XRD (step size 0.002°/s) was inducted to record the selected diffraction peaks. The crystallite size was calculated using the Scherer formula. The surface morphology of the sample is obtained using

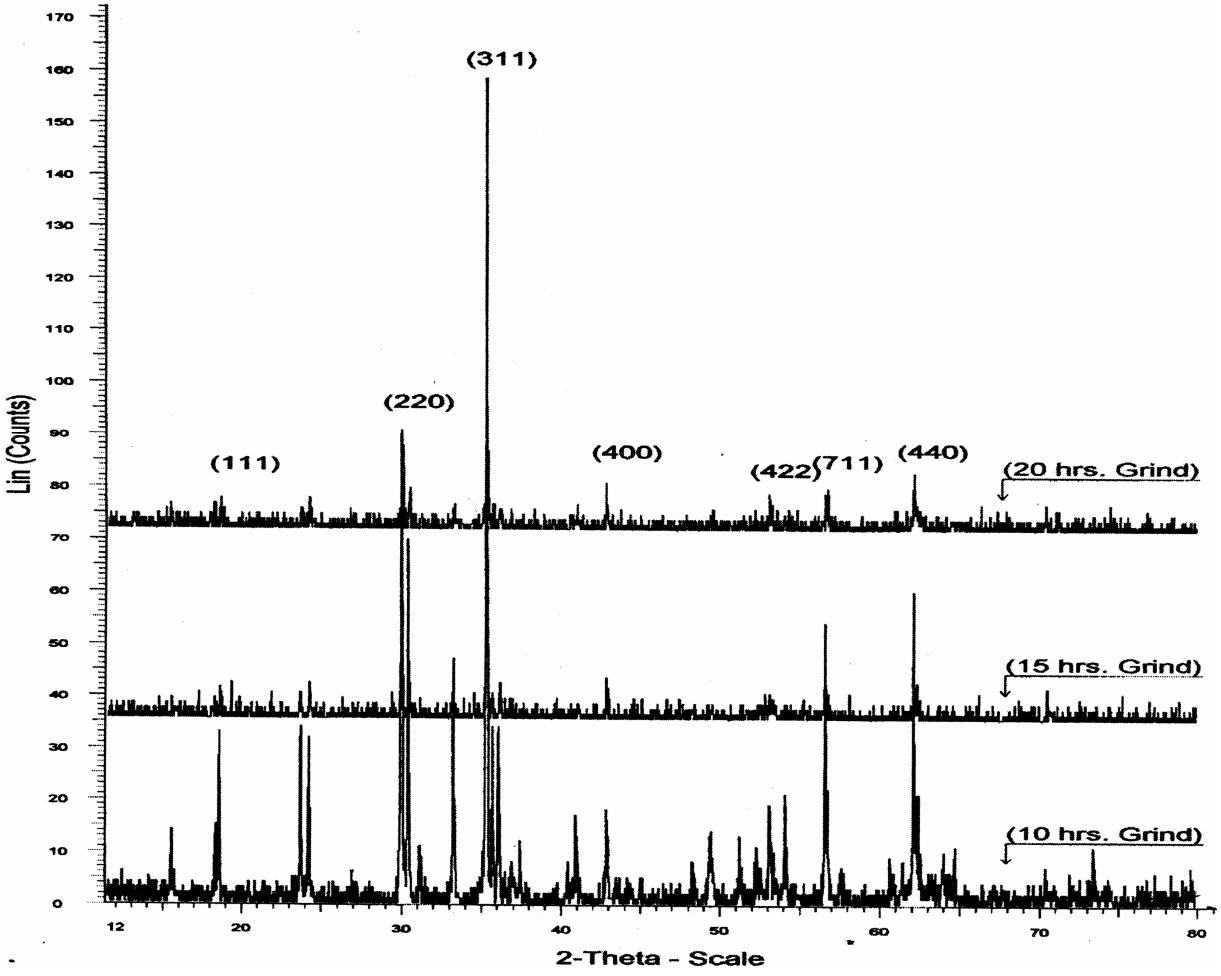

Fig. 1b– X-Ray diffraction patterns of the annealed samples grinded for 10, 15 and 20 h