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Synthesis and Structural Characterization of Nanomanganese Ferrites

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In this paper, the structural properties of nano manganese ferrite are synthesized by using environmental friendly co-precipitation method is reported. The structural parameters such as the lattice constant, average crystallite size (*D*), texture coefficients (*TC*), lattice strain (ε) and dislocation density (ρ) have been determined using X-ray diffraction data.

Keywords: Nanoferrites, Spinel structure, Co-precipitation method, Cubic, Structural analysis.

PACS numbers: 61.46, 75.50Gg, 61.05.cp, 81.20.Fw

1. INTRODUCTION

The semiconductor spinel ferrite will have the chemical formula as AB_2O_4 (where A = divalent metal ion like Ni, Mn, Zn, etc and B = Iron metal ion Fe^{2+} and Fe^{3+}) and many studies have focused on the synthesis of nanomaterials because of extensive applications due to their novel properties. Magnetite and spinel ferrite nanocrystals are regarded as two of the most important inorganic nanomaterials because of their electronic, optical, electrical, magnetic, and catalytic properties, all of which are different from the properties of their bulk counterparts. [1-2] Among spinel ferrites, manganese ferrite ($MnFe_2O_4$) nanoparticles are very important because they have proven to be useful in many magnetic applications, such as recording media devices, drug delivery, ferrofluid, biosensors, and contrast-enhancement agents for MRI technology[3-4]. Also find extensive applications in microwave devices, radar, catalysis and magnetic refrigeration systems [5]. Ferrites are synthesized by several methods like ceramic method [6], chemical methods, solvothermal method [7], microwave combustion method [8], thermal treatment method, sol-gel Method [9-10], EDTA-assisted hydrothermal method [11], oxalate co-precipitation technique [12]. Thus Chemical synthesis routes play a crucial role in designing the final product and also they are better and less cumbersome for the production of fine grained mixed oxide products.

In this present work, we report the synthesis of nano manganese ferrite by environmental frieldly, low cost co precipitation method. The structural information of manganese ferrite powder is done by XRD characterization. The current work also reports the Texture analysis (preferential orientation of planes), x ray density and crystallite size of manganese ferrite.

2. EXPERIMENTAL

2.1 Materials Method

The manganese ferrite in nanosize is synthesized by environmental friendly and low cost chemical co-precipitation method by choosing the AR grade precursors like manganese chloride($MnCl_2 .4H_2O$ 99.5 %, Titan Biotech Ltd.), Iron chloride($FeCl_3$ 98 %, Titan Biotech Ltd.). These precursors are weighed and mixed in desired stiochiometric ratio in distilled water. The mixer is stirred for nearly one hour to get clean solution. Drop wise ammonia (NH_3 25 %, extra pure Sp. gr. 0.91, SDFCL) is added to this solution for maintaining pH and again stirred for one hour. The reddish solution contains precipitate which is filtered to get the manganese ferrite using AR grade filter paper. The paste from precipitate is dried at room temperature and calcined around 800 °C in muffle furnace for 5 hr which will yield the manganese ferrite in nanosize. The process of synthesis of nano manganese ferrite by co precipitation method is schematically as shown in Figure 1. The synthesized nano manganese ferrite is characterized by using Bruker AXS D8 Advance instrument ($\lambda = 1.5406$ Å, detector Si(Li) PSD from STIC, Cochin) for XRD characterization.

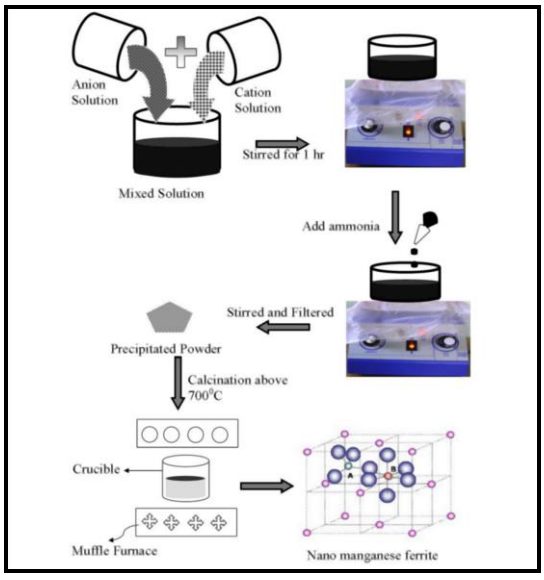

Fig. 1 – Schematic Representation of Synthesis of Nano-manganese ferrite

3. EXPERIMENTAL RESULTS

The cubic spinel structure of nano manganese ferrite is confirmed by X-ray diffraction pattern as shown

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in Fig. 2.The X-ray diffraction pattern is indexed by the JCPDS card no (10-0319, 74-2403). The crystallite size of the nano manganese can be calculated using the Debye-Scherer equation [13] as shown in equation (1).

$$D = \frac{0.9\lambda}{\beta \cos\theta} \quad (1)$$

where λ is the wavelength of used radiation, β is the full width half maximum (FWHM) of diffraction peak, and θ is the Bragg angle.

The average lattice parameter 'a' of nano manganese ferrite found to be 8.63 Å and average crystallite size found to be 42 nm, also the crystallite size for (311) plane at glancing angle(2θ) is 36.83 nm. The measured values of d are in good agreement with the JCPDS card no 10-0319, 74-2403 as shown in Table 1. Table 2 shows the average crystallite size, average lattice parameter, and volume of the nano manganese ferrite.

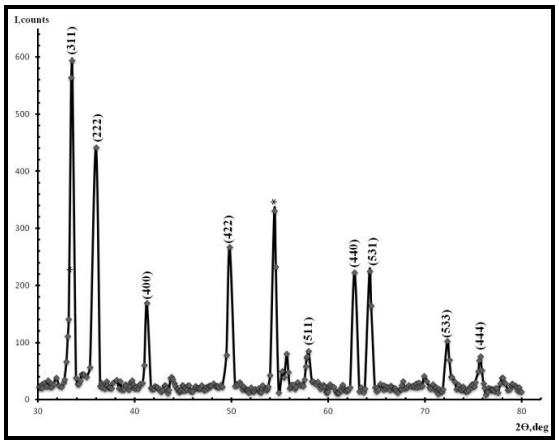

Fig. 2 – X-ray Diffraction pattern of the Nano manganese ferrite

Table 1 – Miller indices and lattice parameter

d(obsverved)	d(standard)	δd	(hkl)	a (Å)
2.66	2.58	0.08	(311)	8.82
2.48	2.46	0.02	(222)	8.59
2.17	2.13	0.04	(400)	8.68
1.82	1.74	0.08	(422)	8.92
1.67	1.64	0.03	(511)	8.68
1.47	1.5	0.03	(440)	8.32
1.44	1.44	0.00	(531)	8.52
1.30	1.3	0.00	(533)	8.52
1.25	1.23	0.02	(444)	8.66

X-ray density (dx) was calculated by using the formula [14] as shown in equation (2)

$$dx = \frac{8M}{Na^2} \quad (2)$$

where M is the molecular weight of the nano manganese ferrite, N is the Avogadro's number and a is the lattice parameter. The X-ray density of nano manganese ferrite found to be 4.76 gm/cm^3.

The lattice strain is defined as the deformation of an object divided by its effective length. This lattice strain is widely utilized in material science which can be calculated by using the formula as shown in the equation (3)

$$\varepsilon = \frac{\beta \cos\theta}{4} \quad (3)$$

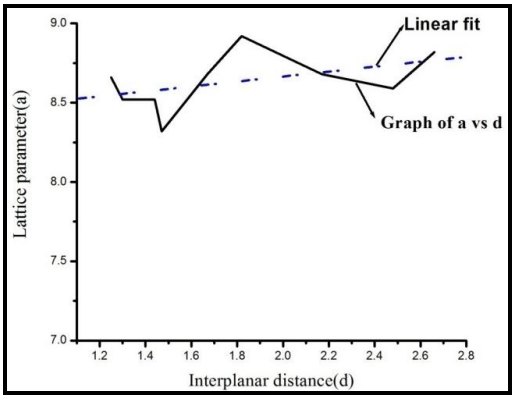

Fig. 3 – Graph of a versus d

Table 2 – Average lattice parameter and Crystallite size

a (Å)	a^3 (Å^3)	D (nm)
8.63	642.74	42

Also the dislocation density can be calculated by using the formula as indicated in equation (4)

$$\rho = \frac{1}{D^2} \quad (4)$$

The lattice strain and dislocation density for nano manganese ferrite found to be 0.082 and 7.37×10^{14} respectively.

Texture is perceived in almost all engineered materials which can have a great influence on properties of materials. Diffraction patterns from samples containing a random orientation of crystallites have predictable relative peak intensities. Texture frequently represents a pole figure, in which a defined axis (crystallographic) from each of a representative number of crystallites is mapped in a stereographic projection [15]. The information regarding degree of orientation of the crystal plane is obtained from the calculation of texture co-efficient (TC) using the formula [16] as indicated in equation (5)

$$TC(hkl) = n\frac{I(hkl)/I_0(hkl)}{\sum I(hkl)/I_0(hkl)} \quad (5)$$

where I and I_0 are the observed and standard intensities and n is the number of reading obtained.

In amorphous solids the crystallites are oriented in random, but each grain in a polycrystalline aggregate normally has a crystallographic orientation different from that of its neighbors that is in polycrystalline like metal oxide the crystals are oriented in particular planes. Hence the texture analysis is carried out by using diffraction technique to know the preferential orientation of the crystallite. So the texture analysis of material is very important factor to study the chemical, mechanical and physical properties of material. From the texture analysis the grain growth is observed at miller indices (311) plane is more dominant ($TC(hkl)$ = 1.56).The detailed $TC(hkl)$ of all plains has been tabulated in Table 3.

Table 3 – Texture Coefficient of all planes of $MnFe_2O_4$

hkl	(311)	(222)	(400)	(422)	(522)	(440)	(531)	(533)
TC(hkl)	1.56	1.38	0.21	0.87	0.64	1.46	1.47	0.45

4. CONCLUSION

Nanosized manganese ferrite has been synthesized by cost effective and simple co-precipitation method. Synthesized nano manganese crystals had a cubic spinel structure with average lattice parameter $a = 8.63$ Å and average crystallite size $D = 42$ nm. Preferential orientation of grains in the (311) plane is predominant and lattice strain and dislocation density for the same plane found to be 0.082 and 7.37×10^{14}. Nano manganese ferrite may find its application in microwave devices, high capacity information storage, water treatment, sensors, inductor cores, and related implementations.

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