IJISET - International Journal of Innovative Science, Engineering & Technology, Vol. 3 Issue 9, September 2016
ISSN (Online) 2348 – 7968 | Impact Factor (2015) - 4.332
www.ijiset.com

L-ascorbic acid assisted sol-gel auto combustion synthesis and characterizations of Ni-Cd ferrite nanoparticles

Vithal Vinayakrao Dhole

[1] Department of Chemistry, B. S .S. Arts & Science College, Makni, Tq: Lohara, Osmanabad, Maharashtra, India

Abstract

Nanoparticles of $Ni_{0.5}Cd_{0.5}Fe_2O_4$ ferrite have been synthesized by sol-gel auto combustion technique using l-ascorbic acid as a fuel. The prepared sample was annealed at 650 ^{0}C for 5 h. Room temperature X-ray diffraction patterns were recorded for the prepared sample to confirm the single phase cubic spinel structure formation. Scanning electron microscope studies was carried out on prepared sample to study the surface morphology. The particle size obtained from XRD and SEM data is of the order of nanometer range. The lattice constant was in the reported range. The magnetic properties were investigated using pulse field hysteresis loop technique exhibited typical hysteresis loop indicating that the sample exhibits ferromagnetic nature. The saturation magnetization and coercivity values were found to be greater compared to bulk counterpart.

Keywords: *Nanoparticles, sol-gel, X-ray diffraction, magnetization.*

1. Introduction

The Ni-Cd ferrites are technologically imperative materials as it possess high saturation magnetization, high resistivity, high stability and low loss energy over a wide range of frequency [1, 2]. In effect, Cd substituted Ni-ferrites are the subject of intensive investigations in the field of fundamental and applied research due to their wide applications in electronic industry. The physical properties of spinel ferrites depend on the distribution of cations over the tetrahedral (A) and octahedral (B) sites [3, 4]. In electronic materials the elastic moduli are of much importance because they shows the nature of binding force in polycrystalline materials and also helps to understand the thermal properties of these materials. The magnetic properties of spinel ferrite originate from the antiferromagnetic coupling between the octahedral and tetrahedral sub lattices. The magnetization results from the difference between the magnetization of tetrahedral (A) and octahedral [B] sites.

The addition of Ni ions in Zn ferrite affects the lattice parameter and influences the Curie temperature. Ni-Zn ferrites have good dielectric properties and have applications in antennas, filters and microwave absorption devices etc [5]. Non-magnetic Cd is similar properties like Zn, Cd is diamagnetic and occupy tetrahedral (A) site. However, an ionic radius of Cd is less as compared to Zn and therefore the solubility of Cd is up to 50%. Therefore, it is fascinating to study Ni-Cd ferrite up to 50% doping [6-9].

The structural, electrical and magnetic properties of Cd substituted nickel ferrite prepared in bulk form have been reported in the literature [10, 11]. However, the structural and magnetic properties of Ni-Cd ferrite prepared by wet chemical sol-gel auto combustion technique are not reported in the literature [12-14].

Sol–gel auto-combustion synthesis method is an simple and convenient method for obtaining nanoparticles of spinel ferrite. Sol–gel technique offers enhanced control over homogeneity, elemental composition and powder morphology. Similar to synthesis methods, synthesis parameters and synthesis conditions also strongly influences the physical and chemical properties of spinel ferrite nanoparticles. In order to obtain materials with desired properties for suitable applications, it is necessary to obtain small and uniform grain size and controlled stoichiometry.

In this study, we report our results on structural, microstructural and magnetic properties of mixed $Ni_{0.5}Cd_{0.5}Fe_2O_4$ ferrite nanoparticles sol-gel auto combustion technique using l-ascorbic acid as a fuel**.**

2. Experimental details

$Ni_{0.5}Cd_{0.5}Fe_2O_4$ nanoparticles were synthesized by sol-gel auto combustion method using analytical grade nickel nitrate ($Ni(NO_3)_2.6H_2O$), cadmium nitrate ($Cd(NO_3)_2.6H_2O$), ferric nitrate ($Fe(NO_3)_3.9H_2O$) and L-ascorbic acid ($C_6H_8O_6.H_2O$) as a fuel. The stoichiometric proportion of metal nitrates to fuel ratio was kept 1:3. The mixed solution was stirred for 15-20 minutes to dissolve completely into distilled water. Ammonia was added drop-wise into the solution to adjust pH value to about 7 and heated at 80 - 90 $^{\circ}$C for 6 h on a hot plate. On the formation of sol-gel, very viscous gel the temperature was further raised up to 120 $^{\circ}$C so that the ignition of the dried gel started and finally powder was obtained. The as prepared loose cobalt ferrite powder was grinded for 30 min and annealed at 650 $^{\circ}$C for 5 h in muffle furnace.

X-ray diffraction (XRD) patterns of all the samples were recorded at room temperature by using a PANalytical

X'pert pro difractometer operated at 40 kV and 30 mA. The diffraction patterns were recorded in the 2θ range 20˚-80˚ with scanning rate of 2˚/min using Cu-Kα radiation of wavelength 1.5406 Á. Magnetic parameters are determined using pulse field hysteresis loop method.

3. Results and discussion

Fig.1 shows the X-ray diffraction (XRD) pattern of $Ni_{0.5}Cd_{0.5}Fe_2O_4$ nanoparticles.

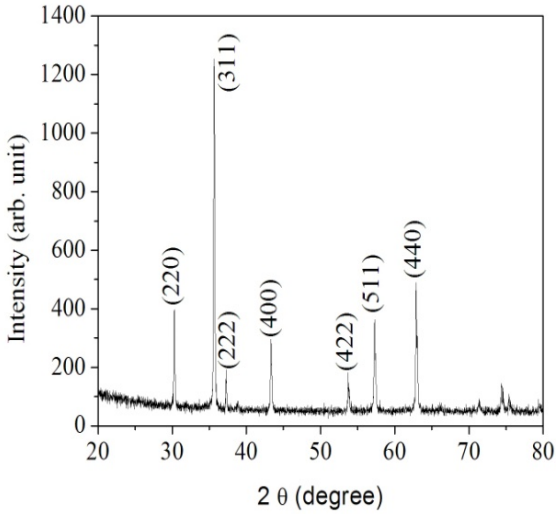

Fig.1. X-ray diffraction pattern for $Ni_{0.5}Cd_{0.5}Fe_2O_4$ nanoparticles

All the Bragg reflections have been indexed, which confirms the formation of cubic spinel structure in single phase. The Bragg's reflections are found to be sharp and intense. The values of lattice parameter calculated from interplanar spacing (d) values and Miller indices are given in table 1. The present value of lattice parameter of Ni-Cd ferrite is in good agreement with the reported value [15, 16]. The average crystallite size was determined from the measured width of the diffraction using Scherrer formula. The particle size obtained from XRD data is found to be 38 nm.

Table 1. Lattice constant, X-ray density and crystallite size from XRD data

Structural Parameters	Values
Lattice constant (a)	8.231 A^o
X-Ray density (ρ_x)	5.299 g/cm^3
Crystallite Size (t)	38 nm

Scanning electron micrograph (SEM) of the prepared sample is shown in Fig. 2.

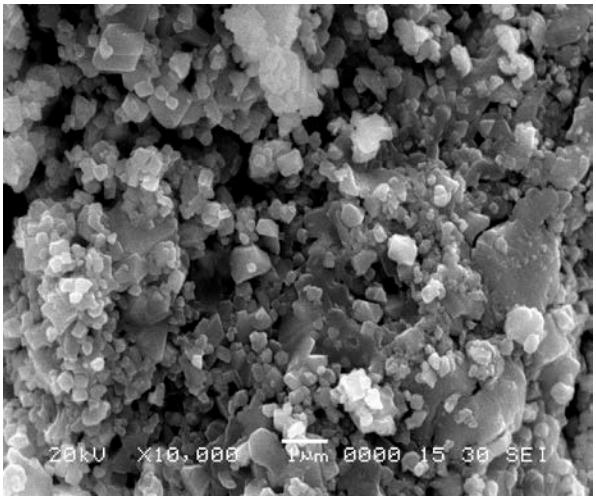

Fig. 2. SEM image of $Ni_{0.5}Cd_{0.5}Fe_2O_4$ nanoparticles

It can be observed that the grains are in nanometer range. The micrograph reveals dense microstructure with developed grains along with few pores.

Fig. 3 shows the magnetization versus field image plot of $Ni_{0.5}Cd_{0.5}Fe_2O_4$ nanoparticles.

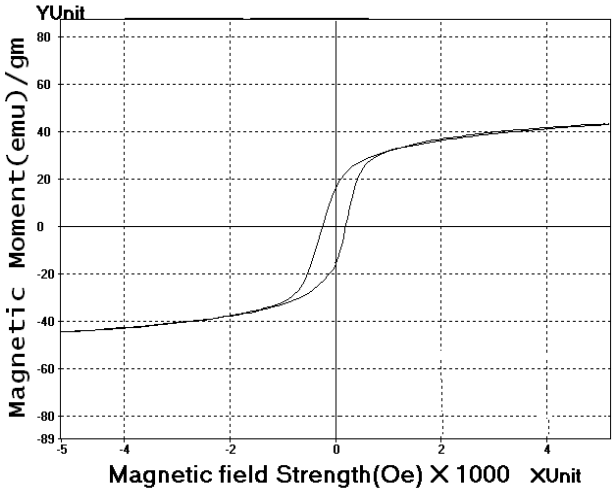

Fig. 3. Hysteresis loop for $Ni_{0.5}Cd_{0.5}Fe_2O_4$ nanoparticles

These plots are used to evaluate saturation magnetization (Ms), remanence magnetization (Mr) and coercivity (Hc). The values of these magnetic parameters are given in table 3. The saturation magnetization values (Ms) are used to calculate magneton number n_B are given in table 4. The observed variation in magneton number was also studied by Neel's theory [17]. According to Neel's theory the magneton number is the difference of magnetic moment of B sub lattice and A sub lattice respectively i.e. $n_B = M_B - M_A$

The calculated value of magneton number is also given in table 4.

Table 3. Magnetic parameters of $Ni_{0.5}Cd_{0.5}Fe_2O_4$

Magnetization Parameters			Magneton number 'η_B' (μ_B)	
Mr (emu/gm)	**Ms (emu/gm)**	**Hc (Oe)**	**Cal.**	**Obs.**
19.82	43.17	220.35	1	0.90

4. Conclusion

The nanocrystalline $Ni_{0.5}Cd_{0.5}Fe_2O_4$ ferrite were successfully prepared by sol-gel auto combustion technique using l-ascorbic acid fuel and AR grade metal nitrates. The X-ray diffraction results showed the formation of single phase cubic spinel structure. The crystallite size, lattice constant and X-ray density are in the reported range. The crystallite size confirms the nanocrystalline nature of the samples. The lattice constant was in the reported range. SEM revealed that the particles are aggregated in spherical shape and in the nanometer range. The values of saturation, remanence magnetization and coercivity show ferrimagnetic nature and are enhanced. The large coercivity (Hc) values indicate the nanocrystalline nature of the present samples.

References

[1]. A. M. Abdeen, J. Magn. Magn. Mater. **185**, 199 (1998).

[2]. B. K. Bammannavar, L. R. Naik and R. B. Pujar, Mater. Sci. an Ind. J. **4(3)**, 160 (2008).

[3]. A. Menakshisundaram, N. Gunasekaran and V. Srinivasan, Phys. Stat. Solidi. (a) **69**, K15 (1982).

[4]. O. H. Kwon, Y. Fukushima, M. Sugimoto and N Hiratsuka, J Phys. **IV**, 165 (1997).

[5]. S.E. Jacobo, W.G. Fano, A.C. Razzitte, Physica B 320 (2002) 261–263.

[6]. S. Akhter, M.A. Hakim, Mater. Chem. Phys. 120 (2010) 399.

[7]. S.S.Suryawanshi, V.V.Deshpande, U.B. Deshmukh, S.M. Kabur, N.D. Chaudhari, S.R. Sawant, Mater. Chem. Phys. 59 (1999) 199.

[8]. S.A.Masti, A.K.Sharma, P.N.Vasambekar, A.S. Vaingankar, J. Magn. Magn. Mater. 305 (2006) 436.

[9]. Saroaut Noor, M.A. Hakim, S.S. Sikder, S. Manzura Hoque, Kazi Hanium Maria, Per Nordblad, J. Phys. Chem. Solids 73 (2012) 227.

[10]. T. Tong, W. Du, Z. Q. Qui, J. C. Walker, J. Appl. Phys. 63 (1988) 4105.

[11]. M. A. Ahmed, N. Okasha, J. Magn. Magn. Mater. 321 (2009) 3436.

[12]. M. A. Hakim, S. K. Nath, S.S.Sikder, K.H.Maria, Journal of Physics and Chemistry of Solids 74 (2013) 1316–1321.

[13]. P. B. Pandya, H. H. Joshi, R. G. Kulkarni, J. Mater. Sci. Lett. 10 (1991) 474.

[14]. S. H. Patil, S. I. Patil, S. N. Kadam, B. K. Chougule, Ind. J. Pure Appl. Phys 30 (1992) 183.

[15]. D. R. Mane, U. N. Devatwal, K. M. Jadhav, Mater. Lett. 44 (2000) 91.

[16]. B. D. Cullity, Moments of X-ray diffraction Adison-Wesley publ. co. Landon (1967).

[17]. L. Néel (1932). Ann. de Phys., 17, 5-105.