International Journal of Engineering Science Invention
ISSN (Online): 2319 – 6734, ISSN (Print): 2319 – 6726
www.ijesi.org ||Volume 6 Issue 10|| October 2017 || PP. 36-39

Structural and Magnetic Properties of Nickel Ferrite Nanoparticles Synthesized by Ball Milling

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Abstract: *Nickel ferrite nanoparticles were synthesized by High Energy Ball milling (HEBM) of the mixture of α-NiO and α-Fe_2O_3 followed by annealing at 1000˚C. X-ray diffraction (XRD), Scanning electron microscopy (SEM), Energy dispersive spectroscopy (EDAX), Fourier transform infrared spectroscopy (FTIR) and Vibrating sample magnetometer (VSM) characterization of the synthesized nickel ferrite nanoparticles was performed. The structural analysis reveals the formation of the single phase compound. The average grain size was estimated by X-ray diffraction technique is 30.13 nm with the lattice constant 8.32Aº. Infrared spectra shows the presence of two strong absorption bands at 603.01 cm^{-1} assigned to tetrahedral and 418.65 cm^{-1} assigned to octahedral complex. The magnetic study at room temperature shows the saturation magnetization and coercivity for milled followed by annealing nickel ferrite nanoparticles were 36.72emu/g and 49.65 Oe. The characterization results of nickel ferrite nanoparticles shows change in the physical parameters depending on the synthesizing technique.*

Keywords: *Nickel ferrite; Spinel structure; HEBM; XRD; SEM; EDAX; FT-IR; VSM*

Date of Submission: 09-09-2017 Date of acceptance: 02-11-2017

I. INTRODUCTION

Ferrites being most important magnetic materials with ferromagnetic properties are extensively studied due to their wide technological importance in magnetic recording media, magnetic storage [1] and contrast agents for MRI [2, 3]. Spinel ferrites being a part of the ferrite family are important ferrite for the field of electronics. Crystal of spinel ferrite possesses the structure of the natural spinels $MgAl_2O4$, determined by Bragg. [4]. Nickel ferrite with the formula $NiFe_2O_4$, consists of a well known inverse spinel structure in which the tetrahedral sites (A) and octahedral site (B) are equally occupied by Fe^{3+} ions while Ni^{2+} ions occupy octahedral sites (B) [5]. Nano size nickel ferrite being soft magnetic material with low saturation magnetization and coercivity is an important material for magnetic applications in telecommunication and power transformers. [6,7]. Nano size nickel ferrite are also useful for gas sensor, humidity sensor [8] and for several catalytic processes [9]. The present study reveals the formation of nickel ferrite nanoparticles by HEBM followed by annealing. As solid state reaction that normally occurs at an elevated temperature are carried out during HEBM at much lower temperature [10] and annealing removes the internal stress introduced due to milling. The results for the structural and elemental analysis by XRD, SEM and EDS, conformation of phase formation by FT-IR absorption bands and magnetic properties characterised by VSM are discussed.

II. EXPERIMENTAL DETAILS

Raw materials used for the synthesis of nickel ferrite nanoparticles are α-NiO and α-Fe_2O_3. These materials was mixed in stoichiometric ratio according to the below equation

$$NiO + Fe_2O_3 \rightarrow NiFe_2O_4$$

The mixture was milled in RETSCH Planetary Ball Mill PM 100 using 10 mm balls for 5 hours at the speed 400 rpm. The obtained powder material was given heating treatment to remove internal stress induced due to milling by annealing it at 1000˚C. The annealed material was grinded into fine powder using agate pestle. The crystal structure of the synthesized nickel ferrite nanoparticles was characterized by X-ray diffractometer Bruker D8 advance with Cu Kα radiation ($\boldsymbol{\lambda}$ = 1.54056 Å) as X-ray source. Qualitative elemental analysis of nickel ferrite nanoparticles was done by Oxford INCA Energy 250, EDS attachment with a Cavl Zeiss EVO MA 15 SEM. Fourier Transform Infrared spectra of the samples were recorded on Vertex 70v spectrometer Bruker in the range of 400-4000 cm^{-1} with KBr as the reference sample. The magnetic properties of the nickel ferrite nanoparticles were investigated by using ADE model EV5 Vibrating Sample Magnetometer (VSM) at room temperature.

III. RESULTS AND DISCUSSION

The X-ray diffraction pattern study for the nickel ferrite nanoparticles synthesized by milling and annealing was done to identify the possible formation of phase. The diffraction peaks for the milled nickel ferrite nanoparticles sample correspond to well indexed reflection planes (2 2 0), (3 1 1), (2 2 2) (4 0 0), (4 2 2), (5 1 1) and (4 4 0) are shown in Fig.1. The respective 2theta values for the reflection planes are 30.35˚, 35.74˚, 37.23˚, 43.44˚, 53.93˚, 57.46˚ and 63.11˚. No impurity peaks are observed in the XRD spectra of as-synthesized sample. The average particle size of the nickel ferrite nanoparticles sample calculated from the maximum intensity peak (311) is 30.13nm, using Debye Scherrer formula: $t = 0.9\lambda/\beta \cos\theta$, where symbols have usual meanings [11]. The lattice parameter calculated as 8.32Å which was comparable lower than bulk material ~ 8.54 Å [12]. The X-ray density calculated for the synthesized nickel ferrite nanoparticles is 5.39 g/cm^3. The inter-ionic cations anions distance [13] calculated for the sample is 1.888Å at tetrahedral sites and 2.032Å at octahedral sites. The radii at tetrahedral sites and octahedral site [13] are 0.5390Å and 0.6813Å respectively. The structural results obtained are in good agreement with the previous research of nickel ferrite synthesized by ball milling. [14, 15].

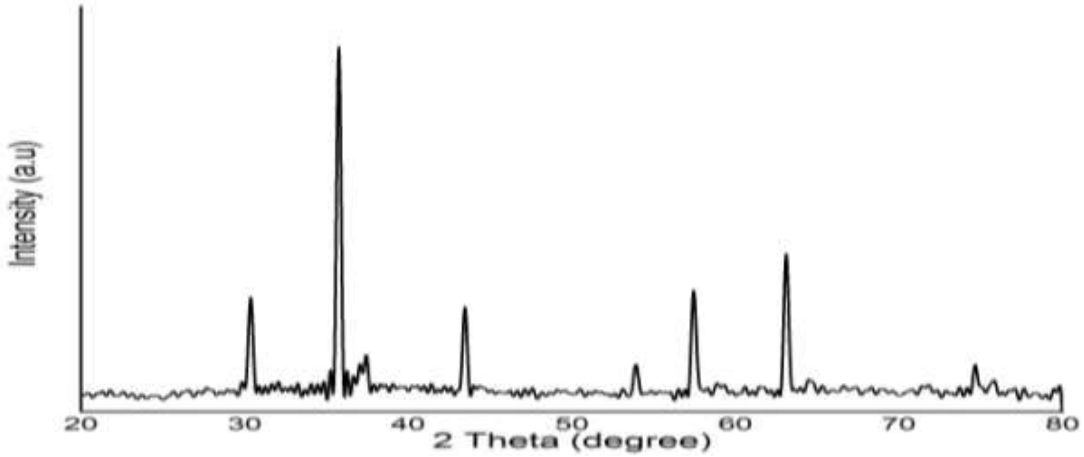
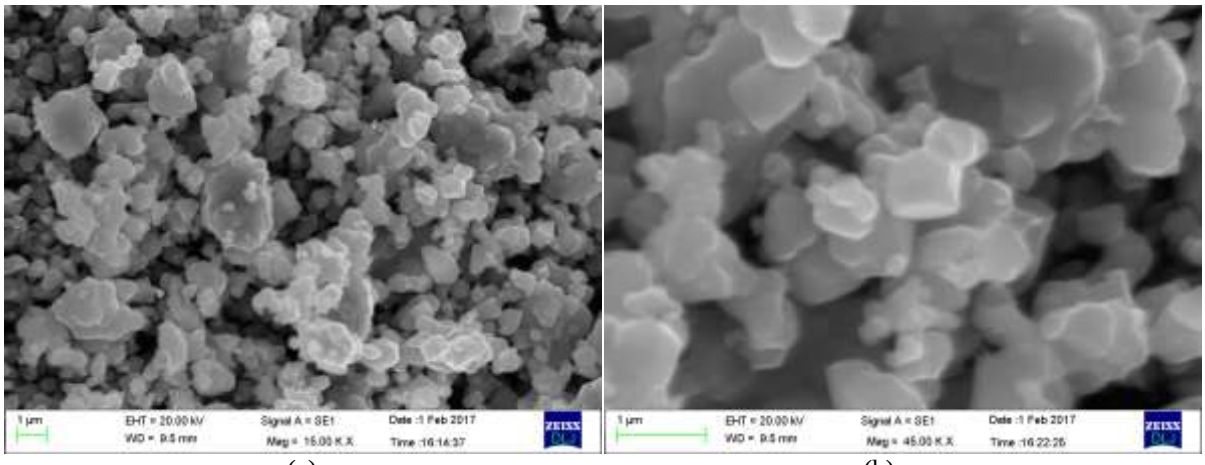


Figure 1: X-ray diffraction spectra of Nickel Ferrite nanoparticle.

The surface morphology characteristics of the synthesized nickel ferrite nanoparticles were investigated using SEM. The Scanning Electron Micrographs (SEM) Fig. 2 shows that the morphology of the particles is very similar. The micrographs exhibit the even distribution of nanoparticles of nickel ferrite. It also indicates the polyhedral shape and narrow size distribution of the particles. As the sample synthesized by milling and annealing results in a very fine powder, so there is formation of soft agglomeration between particles of Nickel ferrite. Fig. 3 shows the EDS pattern for the elemental analysis of the milled followed by annealing nickel ferrite nanoparticles. The elemental analysis confirms the homogeneous mixing of Ni, Fe and O atoms. As there is no impurity peaks observed in the EDS spectrum which reveals the purity of the sample. The compositional stoichiometry of the Nickel ferrites observed by EDS spectra are in good agreement with the stoichiometric calculation.



(a) (b)

Figure 2: SEM images of Nickel Ferrite at (a) 15K magnification and (b) 45K magnification

FT-IR spectra for the milled nickel ferrite nanoparticles are recorded in the range 400 – 4000 cm^{-1} shown in the Fig 4. The infrared spectra indicate the presence of two strong absorption bands. In solids, absorption bands in the range 100-1000 cm^{-1} are assigned due to ions vibration in the crystal lattice. The observed absorption bands within this limit is common feature of spinel ferrite, conforming the formation of spinel structure that consists of two sub-lattice sites, tetrahedral (A) sites and octahedral (B) sites [16]. The higher frequency band ν_1 assigned at 603.01 cm^{-1} corresponds to intrinsic stretching vibration of the metal oxygen (Fe-O) at tetrahedral sites, while lower frequency band ν_2 assigned at 418.65 cm^{-1} are due to metal oxygen stretching vibrations at octahedral sites. The difference in the frequency of absorption of two bands is related to the difference in the bond length of Fe-O at tetrahedral sites and octahedral sites. The shorter Fe-O bond length at A-sites (1.888 Å) leads to high frequency band ν_1 and the bond length at B-sites (2.032 Å) leads to lower frequency band ν_2. The calculated values of the force constant represented by the equation: $K = 4\pi^2c^2\upsilon^2\mu$ [17], where υ is the frequency in cm^{-1}, c is the velocity of light and μ is the reduced mass of Fe^{3+} and O^{2-} ions ($\approx 2.601 \times 10^{-2}$ g), for the tetrahedral site is 2.6450×10^5 dyne/cm and for octahedral site is 1.274×10^5 dyne/cm.

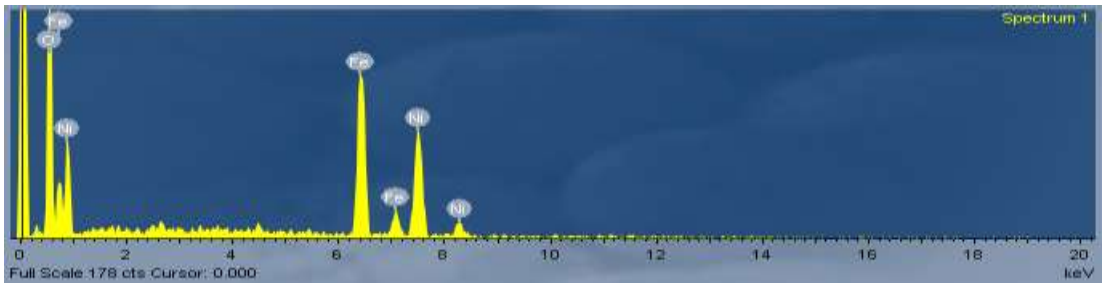


Figure 3: Energy dispersive spectroscopy (EDS) pattern of nickel ferrite nanoparticles.

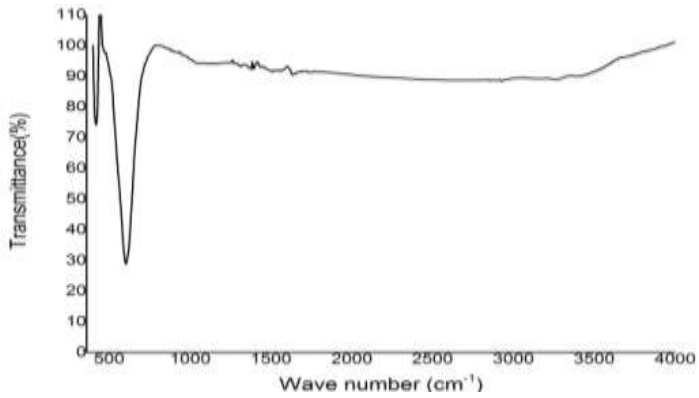


Figure 4: FTIR spectra of nickel ferrite nanoparticles.

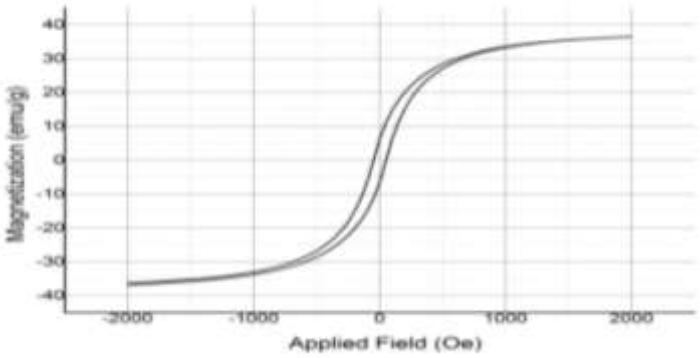


Figure 5: Magnetic hysteresis curve for nickel ferrite nanoparticles measured at room temperature.

Magnetic properties of the synthesized nickel ferrite nanoparticles sample is characterised by VSM (Vibrating sample magnetometer) at room temperature 300K. Shape and size of the nanoparticle highly affects the magnetic properties of the material [18]. Fig. 5 shows hysteresis loop obtained is "S" shape curve that shows

the soft ferromagnetic behaviour of the material. It can be seen from the hysteresis curve that completely saturation position is not reached. Coercivity obtained from hysteresis loop of the milled followed by annealing nickel ferrite nanoparticles sample is 49.65 Oe The investigated value of saturation magnetization for nickel ferrite nanoparticles is 36.72 emu/g which is significantly low as compared to the saturation magnetization of the bulk nickel ferrite (50 emu/g) [19] and reported value for the multidomain bulk nickel ferrite (55 emu/g)[20], while it resembles the reported saturation magnetization value at room temperature [21] and is in good agreement with the previous researches [22,23], the high value of coercivity may be due to high annealing temperature 1000ºC [24].

IV. CONCLUSION

The nickel ferrite nanoparticles have been successfully synthesized by milling and annealing at 1000˚C. The structural investigation confirms the cubic spinel structure of nickel ferrite and the calculated lattice parameter is 8.364 Å. The average crystallite size was found to be 30.13 nm from XRD pattern using Debye Scherrer′s formula. The SEM micrograph reveals the cubical shape of particles. The EDAX spectrum confirms homogeneous mixing of Ni, Fe and O atoms. Infrared spectra shows the presence of two strong absorption bands at 603.01 cm^{-1} assigned to tetrahedral and 418.65 cm^{-1} assigned to octahedral complex. The magnetic study at room temperature shows the formation of soft ferromagnetic material with saturation magnetization 36.72emu/g and Coercivity 49.65 Oe.

ACKNOWLEDGMENTS

The authors acknowledges to DLJ DRDO for providing the facility for SEM, EDS and VSM characterization. The authors are thankful to IIT Jodhpur for providing the service for XRD and FTIR characterization.

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International Journal of Engineering Science Invention (IJESI) is UGC approved Journal with Sl. No. 3822, Journal no. 43302.

H.S.Singh Structural and Magnetic Properties of Nickel Ferrite Nanoparticles Synthesized by Ball Milling." International Journal of Engineering Science Invention(IJESI), vol. 6, no. 10, 2017, pp. 36-39.