Indian Journal of Pure & Applied Physics
Vol. 44, October 2006, pp. 774-776

Synthesis, X-ray diffraction and optical band gap study of nanoparticles of $NiFe_2O_4$

S N Dolia, Rakesh Sharma, M P Sharma & N S Saxena
Department of Physics, University of Rajasthan, Jaipur 302 004

Received 4 October 2005; revised 4 July 2006; accepted 18 August 2006

Nanoparticles of $NiFe_2O_4$ have been synthesized by chemical co-precipitation method. X-ray diffraction pattern confirms the formation of single-phase cubic spinel structure and the lattice constant is 8.2 Å. The energy band gap measurements of nanoparticles of Ni ferrite in pellet form have been carried out by reflection spectra using double beam spectrophotometer. A pellet of nanoparticle ferrite was made under a load of 10 tons. From the analysis of reflection spectra, nanocrystalline Ni ferrite have been found to have energy band gap of 2.5 eV at room temperature.

Keywords: Nanoparticles, Spinel ferrites, Optical band gap, Reflection spectra
IPC Code: H01F 41/30

1 Introduction

Ferrites are superior to other soft magnetic materials for use at high frequency as they show high electrical resistivity combined with useful ferrimagnetic behaviour[1,2]. In recent years, nanocrystalline ferrites have attracted considerable attention, as their physical properties are significantly different from those of bulk[3,4] due to fundamental changes in co-ordination, symmetry and confinement. Superparamagnetism, collective magnetic excitation, low saturation magnetization, enhanced corcivity, metastable cation distribution, etc., are some of the phenomena, which have been observed in nanocrystalline of various ferrites[5-9].

Ni ferrite is extensively used in a number of electronic devices because of their high permeability at high frequency, remarkably high electrical resistivity, mechanical hardness, chemical stability and reasonable cost[10]. $NiFe_2O_4$ is a well-known inverse spinel with Ni^{2+} ions on B sites (octahedral) site and Fe^{3+} ion distributed equally among A (tetrahedral) and B sites. $NiFe_2O_4$ is a typical spin soft-magnetic ferrite and it is extremely interesting to gain its nanocrystalline powder owing to their broad applications[11] such as microwave devices. The properties of nanocrytalline ferrites are size dependent and known to be very sensitive to the processing techenique[12,13]. The nanocrysatlline ferrite is being synthesized by various techniques such as sol-gel[14], hydrothermal[15], ultrasonic emulsion[16], reverse micelle synthesis[17] and combustion[18]. In the present work, we applied chemical co-precipitation method for synthesizing the nanoparticles of $NiFe_2O_4$ and the optical energy band gap of nanocrystalline $NiFe_2O_4$ has been studies.

2 Experimental Details

The nanoparticles of Ni ferrite were prepared by the co-precipitation technique. A mixed solution of 1 M in $NiCl_2.6H_2O$ and 2 M in $FeCl_3.6H_2O$ was slowly poured in NaOH maintaing *p*H of 10. The mixture was slowly heated to 85°C. Oleic acid (5 ml) was added for surafce coating. The solution was made to cool down slowly with continuous stirring. Few drops of HNO_3 were added to precipitate the formed coated particles. The precipitate was washed a number of times with hot distilled water so as to remove NaCl. Finally, water was removed by washing with acetone. This acetone-wet slurry was dispersed in 20 ml of kerosene and heated at 50°C for 5 min. The resulting fluid was centrifuged at 12000 rpm for 10 min. For obtaining the dried particles, portion of the fluid was repeatedly washed with acetone (Fig.1).

X-ray diffraction pattern has been recorded at 300 K using Fe K_α radiation, on a Philips make powder diffractometer PW-1840. The reflection spectrum of nanoparticles of $NiFe_2O_4$ was taken by spectrophotometer Hitachi model U-3400 at room temperature.

The optical reflectance[19] of the pellet was recorded at room temperature in the wavelength range 400-600 nm using a Hitachi (Japan) double-beam-

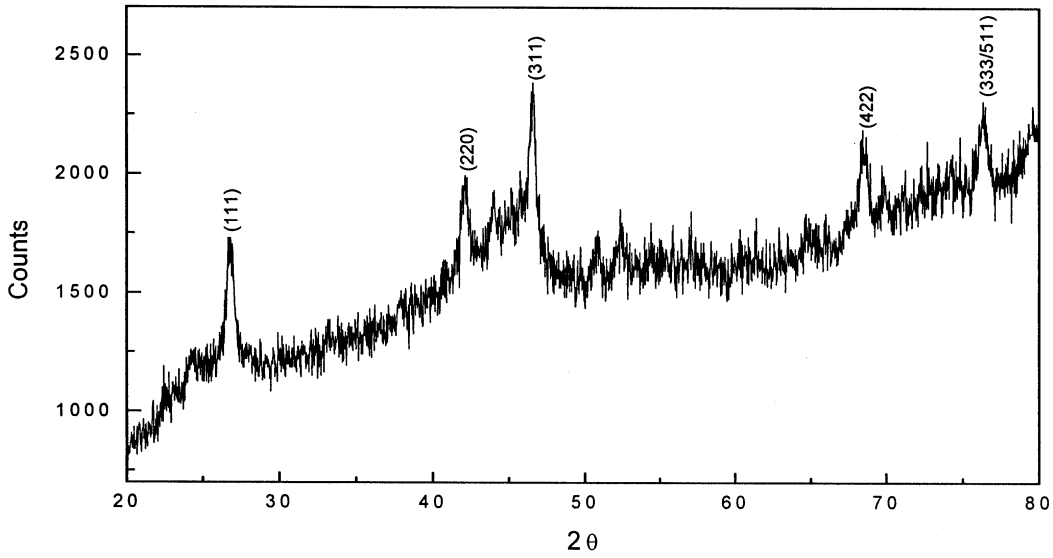

Fig. 1—Powder X-ray diffraction pattern of the nanocrystalline $NiFe_2O_4$ of an average particle size 25 nm

spectrophotometer. In this model, the prism/grating double monochromatic system is used. The optical band gap of the pellet was determined with the help of reflection spectra. Since, it has not been possible to get transmission spectra of the pellet due to high absorption, the reflection spectra for the determination of optical band gap have been recorded. To include the effect of reflection from upper surface of the pellet, a mirror is placed on top of the pellet and reflection spectrum is recorded. According to Tauc relation[20], the absorption coefficient for direct band gap material is given by:

$$\alpha h\nu = A\,(h\nu - E_g)^{1/2} \quad \ldots (1)$$

where hν is photon energy, E_g the band gap and A is constant which is different for different transitions. The absorption coefficient α may be written in terms of reflectance[19] as:

$$2\,\alpha\, t = \ln\,[(R_{max} - R_{min})/(R - R_{min})] \quad \ldots. (2)$$

where t is thickness of pellet and R is reflectance for any intermediate photon energy. A sudden fall in reflectance from R_{max} to R_{min} is observed due to the absorption of light by the material. A graph between the square of $h\nu$ $\ln[(R_{max} - R_{min})/(R - R_{min})]$ (as ordinate) and $h\nu$ (as abscissa) is plotted and a straight line is obtained. The extrapolation of straight line to (hν) axis gives the value of band gap of the material of the pellet.

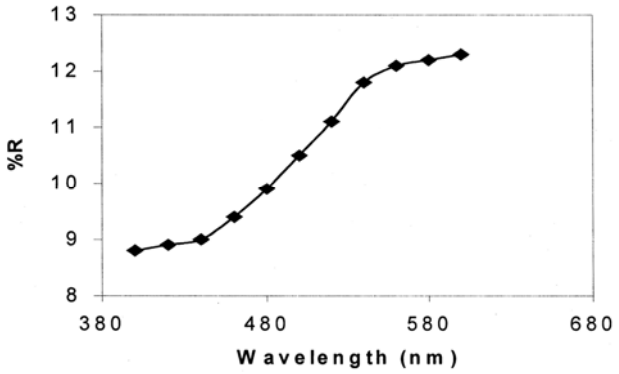

Fig. 2—Reflection spectra of $NiFe_2O_4$ pellet

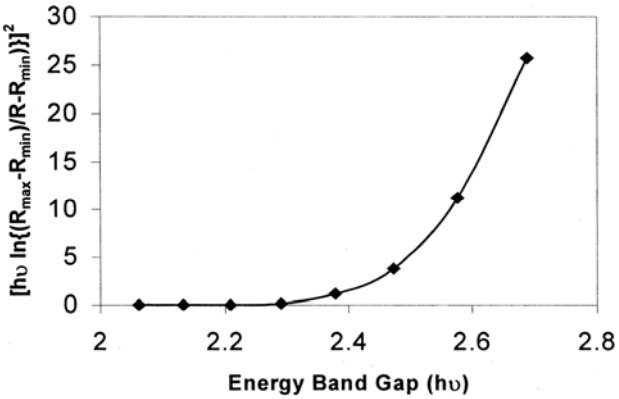

Fig. 3—Band gap determination of $NiFe_2O_4$ pellet. The extrapolation of straight line to ($h\nu$) axis gives the value of energy band gap. The estimated optical band gap is 2.5 eV

3 Results and Discussion

Figure 1 shows indexed X-ray powder diffraction pattern of nanocrystalline $NiFe_2O_4$. The Bragg reflections are indexed in Fe_3O_4 like cubic structure and the estimated cell constant a is 8.2 Å. This confirms that the sample is formed in single phase and the cell constant has slightly decreased with the

introduction of Ni in Fe_3O_4 (a = 8.39 Å). Considerably broadened lines in the XRD pattern are indicative of the presence of nano-size particles. We have used the (311) reflection line in the XRD pattern for obtaining the average particle size with the help of the Debye-Scherrer equation:

$$t = \frac{0.9\lambda}{B\cos\theta_B}; B = \left(B_M^2 - B_S^2\right)^{1/2}$$

where t the thickness (diameter) of the particle, λ the X-ray wavelength (1.9373 Å), B_M and B_S are the measured peak broadening and instrumental broadening in radian, respectively and θ_B in the Bragg angle of the reflection. The calculated average particle size is 25 nm.

Fig. 2 shows the reflection spectra of nanocrystalline $NiFe_2O_4$. It is observed from Fig. 2 that reflection decreases with the decrease in wavelength. A sudden decrease at a particular wavelength is indicative of the presence of optical band gap in this sample. $NiFe_2O_4$ is known to be direct band gap material therefore the Tauc relation is used for the determination of direct band gap. A plot between $[h\nu \ln \{(R_{min}-R_{min})/(R-R_{min})\}]^2$ versus $h\nu$ have been plotted in Fig. 3.

4 Conclusions

XRD pattern of the sample prepared by chemical co-precipitation method shows nanocrystalline nature of the sample. It has been observed that the material prepared is having the lattice constant of 8.2 Å. In highly absorbing samples where transmission spectroscopy cannot be used for determination of energy band gap of samples like in pellets, reflection spectroscopic technique with slight modification has proved to be a useful method. The band gap of the $NiFe_2O_4$ nano-material is 2.5 eV in pellet form.

Acknowledgement

The authors are thankful to Prof. T P Sharma and Dr Subhash Chander, for helpful discussions.

References

1 Slick P I, in: *Ferromagnetic materials*, Vol. 2, ed. E.P. Wohlfarth (North- Holland, Amsterdam), 1980 p. 196
2 Abraham T, *Am Ceram Soc Bull*, 73 (1994) 62.
3 Ozin G A, *Adv Mater*, 4 (1992) 612.
4 Gleiter H, *Adv Mater,* 4 (1992) 474.
5 Albuquerque A S, Ardisson J D, Macedo W A A & Alves M C M, *J Appl Phys,* 87 (2000) 4352.
6 van der Zaag P J, van der Valk P J & Rekveldt M T, *Appl Phys Lett,* 69 (1996) 2927.
7 Fannin P C, Charles S W & Dormann J L, *J Magn Magn Mater*, 201 (1999) 98.
8 Bercoff P G & Bertorello H R, *J Magn Magn Mater*, 213 (2000) 56.
9 Haneda K, *Can J Phys*, 65 (1987) 1233.
10 Ishino K & Narumiya Y, *Ceram Bull*, 66 (1987) 1469.
11 Zhou Z H, Xue J M, Wang J, Chan H S O, Yu T & Shen Z X, *J Appl Phys*, 91 (2002) 6015.
12 Verma A, Goel T C & Mediratta R G, *Second International Conference on Processing Materials for Properties*. The Mineral, Metal & Material Society, 2000, pp. 493-497.
13 Verma A, Goel T C & Mediratta R C, *Mater Sci Technol*, 16 (2000) 712-715.
14 van der Zaag P J, Noordermeer A, Johnson M T & Bongers P F, *Phys Rev Lett*, 68 (1992) 3113.
15 van der Zaag P J, Brabers V A M, Johnson M T, Noordermeer A & Bongers P F, *Phys Rev*, B 51 (1995) 12009.
16 Said M Z, *Matere Lett*, 34 (1988) 305.
17 Rolling B, Happe A, Funke K & Ingram M D, *Phy Rev Lett*, 78 (1997) 2160.
18 Zheng M, Wu X C, Zou B S & Wang Y J, *J Magn Magn Mater*, 183 (1998) 152.
19 Kumar V, Sharma S K, Sharma T P & Singh V, *J Opt Mater*, 12 (1999) 115.
20 Tauc J (Ed.) *Amorphous and Liquid Semiconductors*, Plenum Press, New York, (1974).