International Journal of ChemTech Research
CODEN (USA): IJCRGG ISSN: 0974-4290
Vol.7, No.3, pp 1098-1101, 2014-2015

ICONN 2015 [4th - 6th Feb 2015]
International Conference on Nanoscience and Nanotechnology-2015
SRM University, Chennai, India

Effect of Milling Time of A-Fe_2O_3nanoparticle on the Energy Gap by Planetary Ball Milling

Shilpa.C[1], Deepa.C[1], Aishwariya.R[1], Pradeep.N[1], Chaitra[1].V, Uma.V[1]*

[1]Department of Nanoscience and Technology, Mount Carmel College, Bangalore-560052, India
[2]Department of Nanoscience and Technology, Mount Carmel College, Bangalore-560052, India

Abstract: Synthesis of nanomaterials by a simple, low cost and high yield has been a great challenge since the development of Nanoscience. Hematite nanoparticle (α-Fe_2O_3) has potential applications into many areas such as magnetism, catalysis, electrochemistry, biotechnology, etc. Among all top down approaches, high energy ball milling, has been widely exploited for the synthesis of various nanomaterials, nanograins, nanoalloy, nanocomposites and nano-quasicrystalline materials. Commercial α-Fe_2O_3 powder was used as starting Material. The material was milled for 15, 30, 45 hrs with the speed of 300 rpm. Structural characterization was done using X-Ray Diffraction (XRD) to determine the particle size and orientation. Optical study was carried out by Ultra Violet-Visible Spectroscopy and to determine the magnetic studies has been carried out by Gouy –Balance. Crystallite size of α -Fe_2O_3 decreased with increasing milling time. Optical band gaps of these nanocrystalline α-Fe2O3 samples have been measured from UV-Vis spectroscopy and found to increase, with increasing milling time indicating that the size of the particle is reduced -Quantum confinement effects taken over. The observed values of optical band gaps were in close agreement with the reported values. The magnetic susceptibility showing the interesting results over the different milling times.

***Keywords*:** Iron oxide, Nanoparticles, UV-VIS Spectra, X-Ray Diffraction, Band gap.

Introduction

Iron is widely spread in the nature in different chemical compounds[1].A Great interest in synthesis of Fe_2O_3 because of its environmental friendly properties[2]. Researchers are constantly working on new applications of Fe_2O_3 semiconductor properties are extremely useful in solar energy conversion, photo catalyze, water splitting , media data storage , sensors and also its biocompatible so its extensively used biomaterials for different applications, like cell separation, drug delivery in cancer therapy, magnetic induced hyperthermia, MRI contrast agent, immune magnetic separation IMC and others. Many synthesis methods to have been developed for the growth of Fe_2O_3 nanoparticle such as emulsion Precipitation[3], sol-gel[4], solvothermal[5] method under range of different conditions, here we report the simplest way to prepare the nanoparticle by ball milling technique to find the effect of milling time in energy gap. The crystalline phases were identified by X-Ray Diffraction method (XRD), the absorbance spectra were obtained from UV-VIS spectrophotometer.

Experimental

Planetary ball milling technique is used to synthesis the Fe_2O_3 nanoparticle. Commercial α-Fe_2O_3 powder was used as starting material , where the ball to sample ratio is taken 15:2, the total milling time was 45 hrs at 300 rpm, for every 15 hours sample was taken out for further characterization.

Results and Discussions

Structural determination (XRD) and morphology study

The crystalline structure of the milled Fe_2O_3 powder was determined by means of X-ray diffraction. Figure 1 shows the XRD pattern of milled Fe_2O_3 powder. Fe_2O_3 powder was milled at differnent milling times (15 hrs, 30 hrs, 45 hrs at 250 RPM), ball to powder ratio is 15:2. Structural analysis was carried out by using CuKα radiation, source having wavelength 1.5418 Å. Particle size can be estimated by the Debye-Scherrer formula

$$D_p = 0.94\lambda/\beta_{1/2}\cos\theta$$

Where D is the particle diameter, λ is the X – Ray wavelength, β is the FWHM of the diffraction peak, θ is the diffraction angle. XRD patterns clearly shows only the α-Fe2O3 has the rhombohedral reflections, indicating that there is no other phase transformation has occurred after milled. The orientation peaks observed at (012),(104),(110),(119),(024),(116). XRD peaks for all the three different milling times are almost same. It can be clearly seen all the diffraction peaks are sharp this indicates the sample has crystalline nature and no amorphous phase during the milling conditions. The ball milled Fe_2O_3 showing polycrystalline nature. The orientation peak of (104) is taken for calculation of average crystalline size. Scherer equation calculation shows the decrease of the crystallite size with increasing milling time. The diffraction peaks became broader in 45 hrs compare that 15 hr milled and their relative intensity decreases. This broadening can be attributed to reduction of crystallite size and introduction of defects (microstrain) because of milling process[6]. The force of the impacting balls plastically deforms the powder particles which leads to work hardening and ultimately fracture. This leads to the reduction in particle/crystallite size because of more milling time the powder particles are subjected to high energetic impact with the balls reduction of the particles size in 45 hrs milled powder. The crystalline size for 15 hours is 34.64 nm, 30 hours is 23.16 nm and for 45 hours is 18.46 nm.

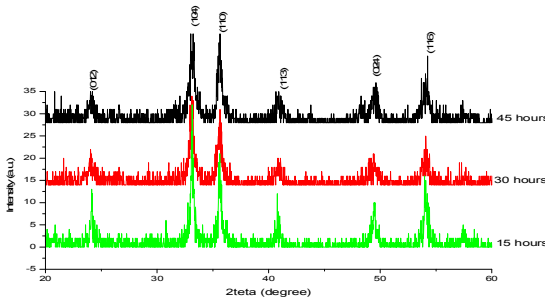

Figure 1: Differnet milling time of XRD paterns

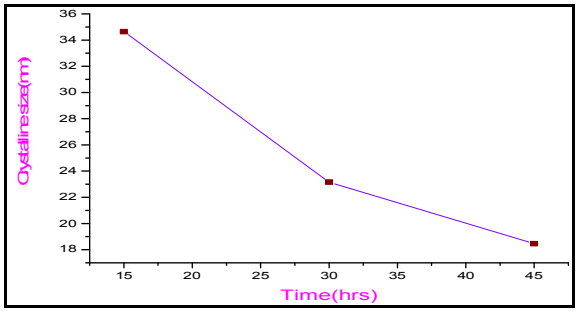

Figure 2: Variation of particle size as a function of milling time

Crystalline size as function of milling time Fig: 2 Particle size was found to decrease with increase in milling time indicating the material moving to the nanoscale.

Optical properties of synthesized Fe_2O_3 nanoparticles

Optical properties of Fe_2O_3 samples were determined through UV-VIS spectrophotometer.The absorption spectra of the samples were recorded after ultra-sonication of the samples in double distilled water. The optical absorbance spectra of Fe_2O_3 nanoparticles at different ball milled powder is as shown in the figure 3.

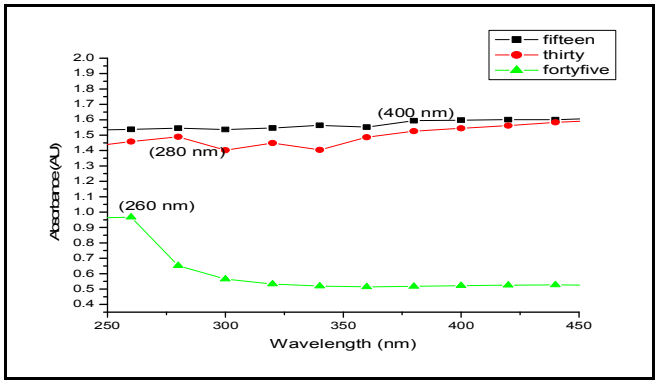

Figure 3: Optical absorbance spectra of Fe_2O_3 nanoparticles at different ball milled powder

The absorption spectra of Fe_2O_3 particles different ball milled hours at room temperature. A typical exciton absorption at 382 nm for 15 hour ball milled powder, An absorption peak at 280 nm for 30 hrs milled powder, and for 45 hours milled powder having absorption peak at 260nm. From above observations we can say that as the milling time increases the absorptionn peak of Fe_2O_3 nanoparticles shifts towards lower wavelength (blue shift) or higher energy indicating the reduction in particle size.

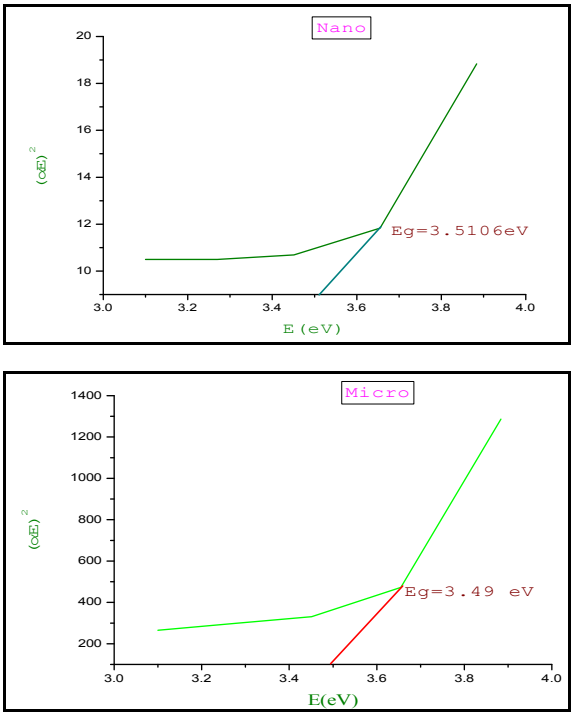

Figure 4 & 5: Shows the graph of αE^2 vs E(eV)

The Energy gap of the nano particle can be determined from the relation,

$$\mathbf{E} = (hc/\lambda)/e$$

Where **h** is the Plank's constant, c is the constant ($c=3x10^8$ m/sec), **λ** is the wavelength in nm, **e**=1.602176565(35)×10−19 C. Absorption co-efficient, **α**=2.303/t(log 1/T) Where **t** is the thickness of the film,

T is the transmittance. $(\alpha E)^2$ is calculated from the above relations; the graph is plotted and Eg is obtained from the graph. The energy gap of the nano sample, Eg=3.5106 eV; which is greater than the micro Fe_2O_3 sample, Eg=3.49 eV. Thus as the particle size reduces from micro to nano level, the Energy gap is increased.

Magnetic susceptibility Studies

In order to measure the magnetic properties of the particles Magnetic susceptibility studies was carried out. Magnetic susceptibility (χ) is defined by the ratio of the induced magnetization (M) to the applied magnetic field (H). The graph of magnetic susceptibility (χ) variation as a function of milling time was recorded (fig.6).

The magnetic susceptibility of the particles was found to increase with milling time; this may be due to increase in the magnetic dipole moment per unit volume indicating the more magnetization in the particles.

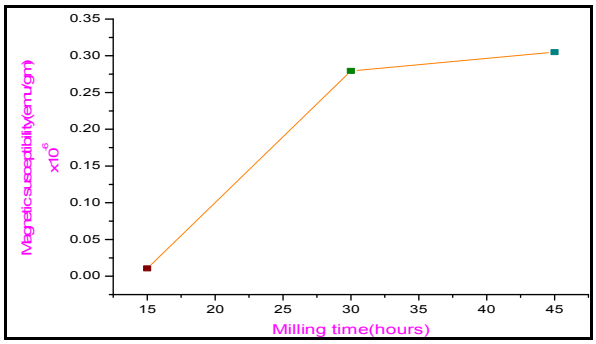

Figure: 6 Magnetic susceptibility (χ) variations as a function of milling time

Conclusion

Nanoparticles of α-Fe_2O_3 with different particle sizes were obtained by planetary ball milling. The material was milled at different milling time (15, 30 and for 45 hrs at 250 rpm) and ball to particle weight ratio of 15:2 was used. Structural characterization was carried out using XRD unit. Particle size for different milling time was determined and it was found to decrease with increasing milling time. The optical absorption was carried out using UV-Vis Spectrophotometer. The absorption peak is moving towards the lower wavelength showing the blue shift. The Energy gap was found to increase with decrease in particle size. The magnetic susceptibility is calculated and it is seen that the susceptibility increases with the reduction of particle size, indicating the material becoming more magnetic in nature. The obtained magnetic susceptibility is positive, so it is Ferro-magnetic in nature.

References

1. Chirita, M., Grozescu, I., Fe_2O_3 Nanoparticles, Physical Properties and Their Photochemical And Photoelectrochemical Application, Chem. Bull. "POLITEHNICA" Univ. (Timişoara), 2009, 54, 1.
2. Qing, W., Zhengcao, L., Zhengjun, Z. and Qin, Z., Facile Synthesis of - Fe_2O_3 Nanostructured Films with Controlled Morphology, Materials Transactions, The Japan Institute of Metals, 2009, 50, 1351-1354.
3. Sahoo, S.K., Agarwal, K., Singh, A.K., Polke, B.G. and Raha, K.C., Characterization of γ- and α- Fe_2O_3 nano powders synthesized by emulsion precipitation-calcination route and rheological behaviour of α-Fe_2O_3, International Journal of Engineering, Science and Technology, 2010, 2, 118-126.
4. Thakur. P.Y., Ram, M.Y., Dinesh, P.S., Nanoscience and Nanotechnology, 2012, 2, 22-48.
5. Basavaraja, S., Balaji, D. S., Mahesh, D. B., Raghunandan, D., Prithviraj, P M S. and Venkataraman, A., Solvothermal synthesis and characterization of acicular α- Fe_2O_3 nanoparticles, Bull. Mater. Sci., 2011, 34, 1313–1317.
6. Saha, S. and Bhunia, A.K., Synthesis of Fe2O3 Nanoparticles and Study of its Structural, Optical Properties.

*****