International Journal of ChemTech Research
CODEN (USA): IJCRGG ISSN: 0974-4290
Vol.7, No.3, pp 1559-1562, 2014-2015

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

Synthesis and Magnetic properties of Pure and Zn doped NiO nanoparticles

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Abstract : Pure and Zn doped NiO nanoparticles (0%, 2%) have been synthesized by a solution phase method employing microwave irradiation using solutions of Nickel acetate and Zinc acetate which are dissolved into a solvent and a fuel. X-Ray diffraction, FTIR spectrum, SEM and Magnetic studies were employed to understand the influence of doping Zn on the structural and Magnetic properties of the nanocrystalline NiO nanoparticles. Magnetic studies confirm the Ferromagnetic nature of NiO. The particle size of the pure and Zn doped NiO were measured as 39nm and 33nm respectively as confirmed from XRD analysis. These studies confirm the presence of NiO and Zn doped NiO nanoparticles with high purity. SEM analysis reveals the average particle size of pure sample to be 100nm.
Keywords: Synthesis and Magnetic properties of Pure and Zn doped NiO nanoparticles.

Introduction

Over recent years increasing attention has been focused on the production of novel nanostructured metal oxide material. NiO received a considerable attention due to its wide range of applications such as smart windows, spin valves, giant magneto resistance (GMR) sensor, Solar cell etc[1]. NiO is an anti ferromagnetic oxide semiconductor with p-type conductivity due to its wide band gap energy range from 3.6 to 4.0 eV[2]. Transition Metal doped NiO samples have been studied mainly for their dielectric and magnetic properties. However, the major focus has been confined to the evolution of magnetic property in NiO with Transition metal doping. Doping of transition metal elements at Ni site modifies the magnetic, electrical, optical and to some extent structural properties and open up the possibility of exploiting this material for DMS functionality.

The structural property of nanoparticle is closely related to the preparation techniques. Several methods have been used and developed for synthesizing crystalline oxide powders in nanoscale dimensions. In many of them, the main objective is to reduce the cost of chemical synthesis and to produce materials for technological applications. Among various methods, the preparation of NiO nanoparticles through solution phase method employing microwave irradiation opens a new view for chemists. Since, microwave methodologies are unique in their ability to be scaled up without suffering thermal gradient effects, providing a potentially, industrially important improvement in nano crystal synthetic methodology over heating not only enhances the rate of

formation, it also enhances the material quality and size distributions [3]. The properties of NiO are strongly modified by doping with transition metal oxides. Here, we report Structure, optical and magnetic properties of Zn doped NiO nanoparticles.

Experimental

Samples of nominal Zn doped NiO (0.0, 0.20) were prepared by the solution phase method employing microwave irradiation. High purity (MERCK) Nickel acetate $Ni(CH_3CO_2).2H_2O$, Zinc acetate $Zn(CH_3CO_2).2H_2O$, Urea(CH_4N_2O) were used as precursors. Ethylene glycol used as the solvent. Nickel acetate and urea were weighed and dissolved into Ethylene glycol and the process was accompanied by magnetic stirring for an hour. The resultant solution was microwave treated until the solvent evaporates completely. The final product was washed several times with deionized water and acetone in order to remove any possible ionic remnants, collected by filtration and then dried in an oven at 70°C and further in furnace at 300°C. Similarly Zn doped NiO nanoparticles were also prepared by the same procedure using Zinc acetate as the precursor. High purity samples are synthesized.

The synthesized nanoparticles were characterized by X-ray powder Diffractometer, Fourier Transform Infrared Spectroscopy, SEM and VSM.

Results and Discussion

Figure.1 shows the XRD patterns of pure NiO Sample1 and Zn doped NiO sample 2. The existence of strong and sharp peaks located at the 2θ values 37.16°,43.29° and 62.8° corresponds to (1 1 1),(2 0 0),(2 2 0) planes respectively indicates the formation of pure NiO nanoparticles.[4,5]. The diffraction peaks of NiO matches with JCPDS data[JCPDS file: 895881]. The existence of new peak at 43.2° corresponds to doped Zn. These diffraction peaks matches well with JCPDS data [JCPDS file: 653358, 894721].

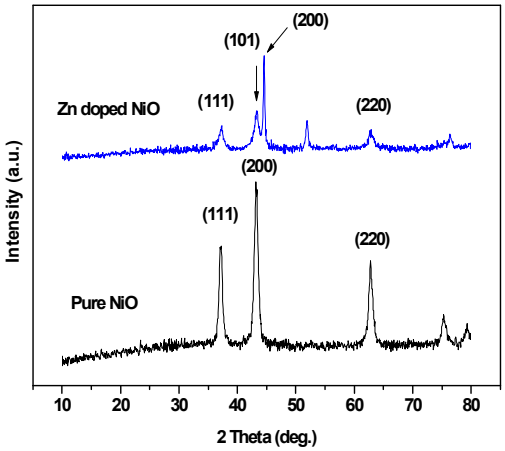

Fig 1. X-Ray Diffraction Pattern for Pure and Zn doped NiO nanoparticles.

The average particle size of the pure NiO nanoparticles was determined using Debye Scherrer equation:

$D=K\lambda/\beta COS\theta$

Where K is a constant equal to 0.39, β is the full width half maximum height of the diffraction peak at an angle θ and λ is the wavelength. The particle size of synthesized NiO nanopowders for pure NiO is around 39 nm. For Zn doped NiO samples the particle size decreases upto 33 nm. The particle size gets reduced when the doping ratio increases.

SEM image of pure NiO nanoparticles is shown in Figure 2. It reveals that pure sample consists of nanoparticles with non homogeneous size and shape with average size around 100 nm. This value mismatches with the particle size estimated by X-ray diffraction. This may be due to agglomeration of particles. The agglomeration can be induced by densification resulting from the narrow space between the particles due to the uniform distribution of oxidized metal anions in the three dimensional polymeric network structure. The

nanoparticles tend to agglomerate during synthesis or delivery process due to their high surface area and surface energy.

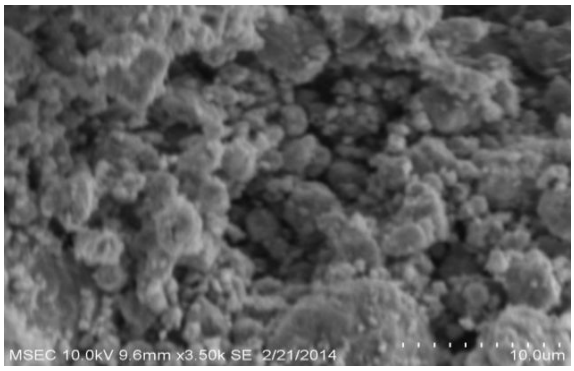

Fig.2. SEM Image of pure NiO nanoparticles

In the FTIR spectra, the main bands were observed at ~ 3600,1632,1378,1030,830 and 414 cm^{-1}. The band at ~3600 represents stretching of O-H group. The band 1632, 1378 cm^{-1} represents carboxylate group of NiO. The band at 1030 cm^{-1} represents C-O group in stretching mode[6,7]. The band at 830 cm^{-1} corresponds to bending vibration of NiO[6] [Figure.3]

In the FTIR spectra of Zn doped NiO, the main bands were observed at ~ 3600,1627,1380,1036 and 447 cm^{-1}. The NiO bands are shifted to lower frequency region because of doping of Zn. The band 1632 cm^{-1} shifted to 1627 cm^{-1}. The band 1378 cm^{-1} shifted to 1380 cm^{-1}. The band 1030 cm^{-1} shifted to 1036 cm^{-1}. The band at 830 cm^{-1} disappears when it is doped with Zn. The band 447 cm^{-1} represents the bending vibration of Zn nanoparticle[8].

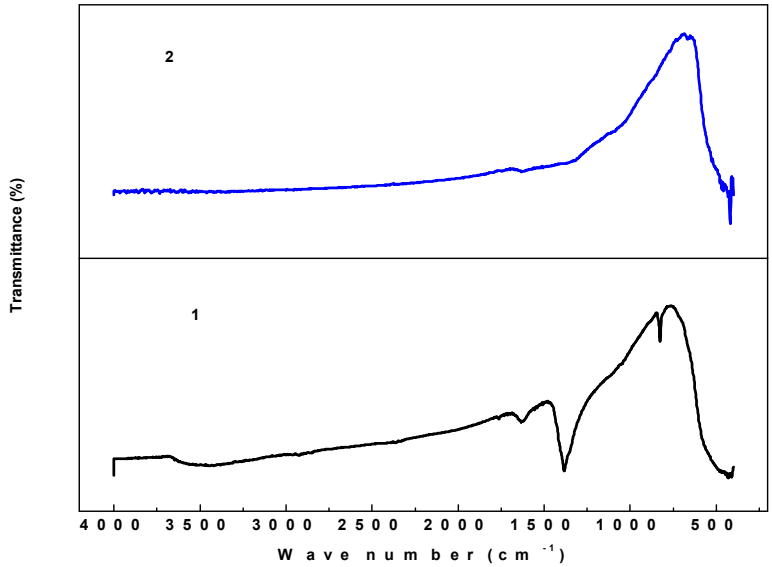

Fig. 3. FTIR Spectra of Pure and Zn doped NiO nanoparticle

Figure 4 shows a typical hysteresis loop obtained at room temperature for pure and Zn doped NiO nanoparticles. The saturation magnetization (M_s) retentivity (M_r) and coercive field of the pure NiO were found to be 0.23 emu/gm, 35.57 x 10^{-3} emu/gm and 152.01G respectively. The saturation magnetization (M_s), retentivity (M_r) and coercive field of the Zn doped NiO were found to be 14.318 emu/gm, 2.721emu/gm and 151.58G respectively. The Squareness ratio (SQR=M_r / M_s) is an important assessment of the quality of the magnetic materials. The SQR for NiO was found to be 0.152 indicating that the pure NiO do not have the properties of recording medium[11]. Magnetization measurements in these cases reveals that Zn doped NiO nanoparticles shows the room temperature ferromagnetic behaviour and increases with Zn doping[10].

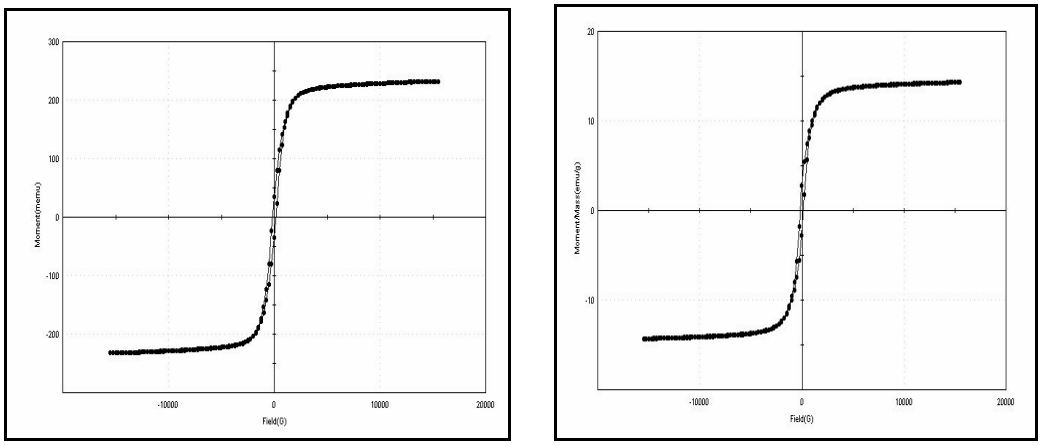

Fig.4. Hysteresis curve measured at room temperature for pure and doped samples

Conclusion

NiO Nanoparticles were successfully synthesized by the solution phase method employing microwave irradiation using Nickel acetate, urea and ethylene glycol. XRD, FTIR, SEM and VSM studies are carried out .The FTIR spectra indicate the formation of Pure and Zn doped NiO nanoparticles. The XRD spectrum shows the samples are in single phase. The results reveal the formation of high purity NiO and Zn-NiO nanoparticles. The pure NiO particles are having FCC structure. The particle size of synthesized NiO nanopowders for pure NiO is around 39 nm. For Zn doped NiO samples the particle size decreases upto 33 nm. The magnetic studies reveals the room temperature ferromagnetic behaviour of NiO nanoparticles. The prepared Samples can be used as a soft electro-magnetic materials in constructing transformers and cathode materials in batteries.[11]

References

1. Gokul, B., Matheswaran, P., Structural and dielectric properties of NiO nanoparticles. J Non- cryst solids., 2013, 363: 161-166.
2. Anandan, K., Rajendran, V., Structural,optical and magnetic properties of well-dispersed NiO nanoparticles synthesized by CTAB assisted solvothermal process. Nanosci nanotechno., 2012, 2: 24-29.
3. John Kennedy Vethanathan, S., Perumal, S., Structural and magnetic properties of nickel and cobalt doped ZnO nanoparticles synthesized by solvo thermal route. Int J sci ind res., 2013, 3: 856-865.
4. He, J., H, Yuan., S L, Tian., Z M, Yin., Y. S., Li P., Wang Y. Q., Liu, K, L., Yuan, S., J,Wang, X. L., Liu, L., Magnetic properties in Fe-doped NiO Synthesized by co-precipitation. J mag magnet mater., 2008, 320: 3293-3296.
5. Kashani, M., Motlagh, M., Youzbashi, A. A., Synthesis and characterization of nickel hydroxide/oxide nanoparticles by the complexation-precipitation method. Int J phys sci., 2011, 6: 1471-1476.
6. kwanruthai Wongsaprom., SantiMaensiri., Synthesis and room temperature magnetic behavior of Nickel oxide nano crystals. Chiang mai J Sci., 2013, 40: 99-108.
7. Harish Kumar., Renu Rani., Structural and optical characterization of ZnO nanoparticles-synthesized by microemulsion route. Int lett Che Phy astro., 2013, 14: 26-36.
8. Sangeetha Nagarajan., kumaraguru., Arumugam., Karuppasamy., Extra cellular synthesis of Zinc oxide nanoparticle using sea weeds of gulf of Mannar, India., J NanoBio Tech., 2013, 4: 11:39.
9. Manna, S., De, S. K., magnetic properties of Li and Fe co-doped NiO., Solid State Commun., 2009, 149: 297-300.
10. Mallick, P., Mishra, N. C., Evolution of structure, Microstructure, Electrical and Magnetic properties of Nickel oxide(NiO) with Transition metal ion doping. Am J Mater. Sci., 2012, 2: 66-71.
11. Nandapure. B. I., Kondawar. S. B., Salunkhe. M. Y., Nandapure. A. I., Magnetic and transport properties of conducting polyaniline/nickel oxide nanocomposites., Adv Mater lett., 2013, 4: 134-140.

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