Article

SYNTHESIS AND CHARACTERIZATION OF Gd-DOPED ZINC OXIDE NANOCRYSTALLINE POWDERS

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Abstract

One-step aqueous solution method was used to synthesise the ZnO and Gd-doped ZnO nanocrystals. X-ray Diffraction (XRD), Scanning Electron Microscopy(SEM) analysis have been used to characterize these nanocrystals. The XRD studies revealed that the ZnO and Gd -doped ZnO had wurtzite structure. The analysis of composition by EDX confirmed the presence of Gd in these nanocrystals. The quality of the syntheised ZnO and Gd -doped ZnO nanocrystals was good. At room temperature, these ZnO and Gd -doped ZnO nanocrystals show diamagnetism. Pure and Gd doped ZnO nanocrystals were prepared fromGd the mixture of zinc acetate, Ga-acetate and potassium hydroxide aqueous solutions. The Gd-doped ZnO nanocrystals of size 100-170nm were successfully synthesized and characterized.

1. Introduction

Nanomaterials have attracted tremendous interest in recent years because of their unique physical and chemical properties, which are highly different from those of either the bulk materials or single atoms [1, 2]. It is a highly challenging work to develop preparation methods that are scalable, cost-effective and environment friendly [3]. Several methods are available for growing nanomaterials in particular ZnO. Some of them are hydrothermal method, atmospheric pressure PVD method, non-hydrolytic route method, wet chemical method and refluxing technique,etc. The ultra-fast synthesis route for the preparation of Gd-doped ZnO nanocrystals is developed in order to save the time. Growth of transition metal and Rare-earth doped wide band gap semiconductors, is now gaining interest. Nano-structured ZnO doped with different rare-earth elements is becoming increasingly important in the present times because of its distinguished properties such as electrical, optical, thermal and magnetic finding potential application as Bio-medical sensors, transducers and catalysts. Many researchers have also worked in this direction.

In the present work, pure Gd doped ZnO nanocrystals were prepared by a simple, quick and economic method using chemical synthesis route and characterized using FTIR and SEM. The results obtained are reported herein.

2. Materials and methods

All Analytical Reagent (AR) grade reagents are used in this sysnthesis. Zinc Acetate ($(CH3COO)_2Zn.2H_2O$) and urea (NH_2COH_2) were taken in 1:3 molecular ratio and dissolved in deionsied Water. Few drops of Ethylene glycol is added to the solution. This solution is taken in a Borosil beaker (200ml) and introduced into a muffle furnace. The furnace temperature is raised to 400^{o}C from room temperature with a heating rate of 15^o/min. The furnace is kept at 400oC for 45 minutes and the beakers were taken out and pale coloured powder is taken out from the beaker with the help of glass rod. This powder is transferred to pre-heated Silica crucible at 400^{o}C and introduced into another furnace kept at 900^{o}C. The powder is combusted at 900^{o}C for 30 minutes. The crucible is taken out and cooled to room temperature. The powder in the crucible is found to increase its volume and this foamy powder is taken out with the help of the glass rod and used for characterization. The Gd doped ZnO nanocrystals were synthesized by adding 1 mol% of Praseodymium carbonate to the Zinc carbonate and Urea solution in the initial stages of synthesis.

Using an automated X-ray powder diffractometer with monochromated CuKα radiation (λ= 1.54056Å) the powder X-ray diffraction data were collected for all the samples and they were compared with the available literature data for the identification of the sample. Using the Scherrer formula [7] the grain sizes were determined.

Synthesized ZnO nanocrsytals were initially characterized by Fourier-transform infra red spectrum using Bruker FTIR spectrometer. The spectrum was recorded in mid-IR region of 400-4000cm-1 with 16 scan speed, using attenuated total reflectance (ATR) technique. Scanning electron microscopic (SEM) analysis was done using Zeiss, EV-18 model scanning electron microscope. A thin film of the sample was prepared on a carbon coated copper grid by placing small amount of the sample on the grid. Then it was allowed to dry using mercury lamp for 5 min. Energy Dispersive X-ray analysis (EDX) was carried out on Zeiss, EV-18 model. The peaks obtained from EDX gives the element composition of the sample. The prepared samples were subjected to and magnetic studies also.

3.Results and discussions

3.2 SEM analysis

The SEM images of the samples the samples were presented in Figure 1. The SEM images clearly show nanocrystalline behavior of the synthesized sample. The nanocrystals of the size 100nm to 350nm are clearly distinguishable.

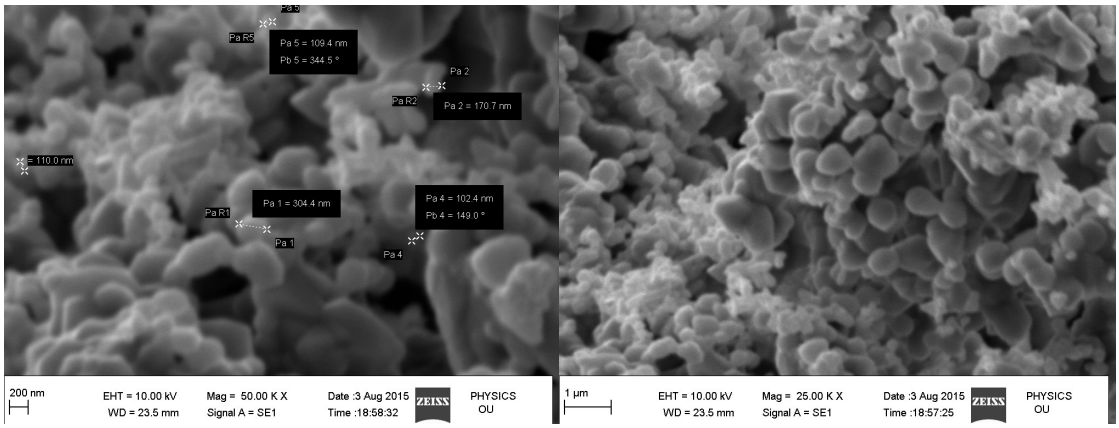

Fig.1. SEM images of Gd doped ZnO nanocrystals

3.3 EDX analysis

The EDX spectra show the purity of the material and the complete chemical composition of synthesized Zinc nanoparticles. In the present synthesis EDX analysis shows 91% to 94% purity of the Zinc in the zinc nanostructures developed in this study. EDX analysis indicates the presence of Pr in the present samples.

3.4 XRD analysis

The peaks obtained matched more appropriately with the hexagonal phase [structure] for Zinc Oxide (JCPDS Card No:36-1451). Almost all the peaks are dominant indicating the crystalline nature of the prepared samples. However, broadening related to size reduction exists. The average grain size of the samples was found by using Scherrer.

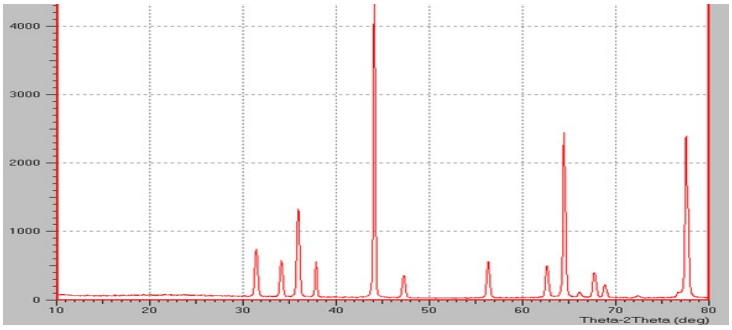

Figure.2. XRD of Gd doped ZnO nanocrystals.

It is revealed here that all the prepared ZnO nanocrystals are in the nano regime with average grain size ranging from 15 to 17nm (The Miller indices are calculated from peak angles and corresponded to hexagonal structure. The lattice distance dhkl (Å) is estimated from Bragg's law. The lattice constants are estimated as following: a=b=3.287 Å, c=5.13 Å. This is in consistency with the reported values indicating the variation for a=b from 3.27 Å up to 3.41 Å and c from 5.01 Å up to 5.37 Å.

3.5 Particle Size analysis

The results of the particle size analysis clearly indicates the nanocrystalline size to be lying between 100 nm to 170 nm, with the maximum particles to be with a size around 120nm.

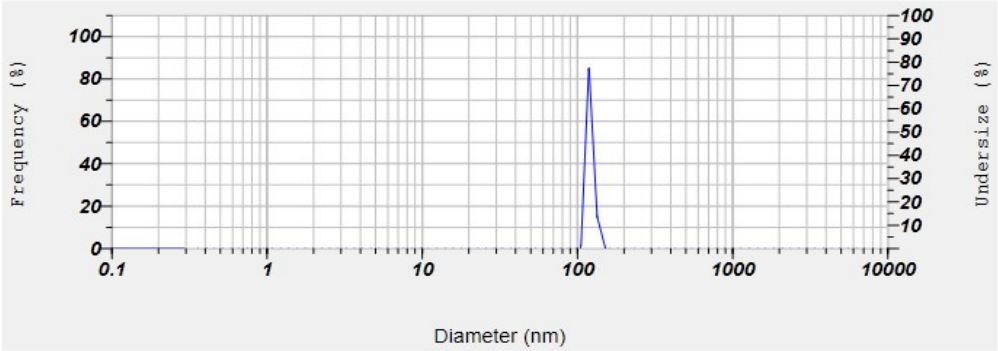

Figure.3. PSA of Gd doped ZnO nanocrystals.

3.6. FTIR spectra

The FTIR spectra of the Gd doped ZnO nanocrystals is presented in Figure.4. The peaks are almost coinciding with the reported peaks in literature for ZnO except few. These are exactly matching with the FTIR bands of GdO.

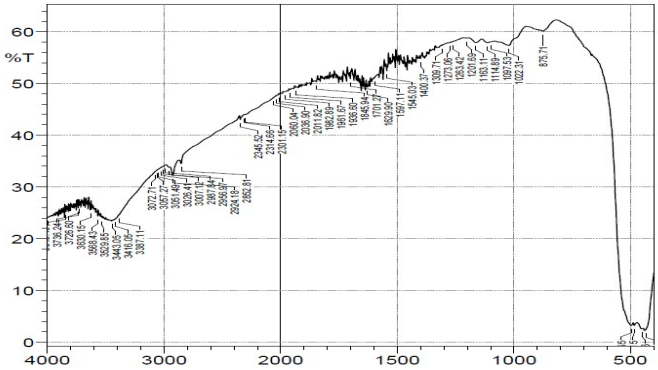

Figure.4. FTIR spectra of Gd-doped ZnO nanocrystals.

4. Conclusions

It is concluded that the method adopted in this synthesis is quick and time saving. It is also low-cost and eco-friendly. Gd-doped ZnO nanocrystals were prepared by the simple method.The XRD analysis indicates thatall the prepared ZnO nanocrystals are in hexagonal phase structure. The results indicate that the Particle size is around 120 – 170 nm.

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