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Synthesis and characterization of ZnO nanoparticles by sol-gel method.

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Abstract

Zinc oxide nanoparticles were prepared by sol-gel technique via two different approaches having by employing zinc sulphate and sodium hydroxide. In the first approach, NaOH was added dropwise with regular interval (ZnO-A) whereas in the second approach NaOH was directly mixed with ZnSO4 solution (ZnO-B). The physical properties of the synthesized nano-particles were studied by X-ray diffraction technique and scanning electron microscope to investigate the structure and morphology of the material, respectively. It was observed that the both the grown ZnO particles revealed the crystalline properties. From the Debye-Scherrer formula , the particle size of ZnO-A was found to be 10nm whereas for ZnO-B it was found to be 7.7 nm. The SEM morphology exhibited the grown particles have uniformity. The synthesized ZnO particles could be used for various opto-electronic devices.

Keywords: Debye-Scherrer Formula, sol-gel, FWHM, Whatman filter paper.

Introduction

Due to diverse technological applications, ZnO has been an active area of research for more than half a century. It has many interesting optoelectronics properties such as wide band gap (~3.37 eV), a large excitation binding energy of 60 meV and high dielectric constant. Therefore, it can be used in the fabrication of electronics and optical devices such as UV/blue laser (Jhonson*et al* 2001), light-emitting diodes (LEDs), Solar Cells, and Organic light-emitting diodes (OLEDs). The size dependent optical properties of this material are very interesting and it can also be tailored with annealing temperature.

Because of these properties, it can be used for the wide range of applications like fabrication of switching element, transistors (Arnold et al 2002) and detectors. It is transparent to the most of the solar spectrum, therefore widely used for window material in solar cells, optical waveguide, light modulators and optical sensors. Therefore the controlled synthesis of good quality ZnO nanostructures, nano crystals (Singh & Gopal 2008a), nano wires, nano belts and other nano architecture are very important. Several routes are employed to synthesis the ZnO nano materials particularly solvothermal (Dev*et al* 2006), thermal evaporation (Pan *et a*l 2001, Kong *et a*l 2004), solid state pyrolysis (Wang *et al* 2003), chemical vapour deposition (Fay *et al*2005; Xiang *et al* 2007), molecular beam epitaxy (Agashe*et al* 2004)

and laser ablation (Singh *et al* 2008) etc. These technologies are very expensive and difficult to use for large area fabrication. Therefore, to overcome these difficulties, we have adopted the low-cost sol-gel technique to grow the ZnO nanomaterials.

In this paper we report the characterization of ZnO nanomaterial synthesized by ZnO different method using hydroxide route. Structural and surface morphology properties have been studied by using X- Ray Diffraction and Scanning Electron Microscope techniques.

EXPERIMENTAL

ZnO is prepared by conventional precipitation method using sodium hydroxide and zinc sulphate solutions. Calcinations of zinc hydroxide gives zinc oxide in powder form. For the preparation of zinc hydroxide, sodium hydroxide solution is mixed with sodium suphate solution in two ways, in the first approach (i.e. ZnO-A) sodium hydroxide is mixed drop wise with zinc sulphate solution and for second approach (ZnO-B) it has been mixed in one-shot with the same.

First of all homogeneous solution of NaOH is prepared by dissolving 1.6 g of NaOH (Merk Specialties Private Ltd, India) in 40 g of distilled water. The pH value of NaOH was found 11.57. The recorded humidity and temperature during experiment was found 29% and 34.5^0C respectively.

Similarly, a homogeneous solution of $ZnSO_4$ (Chemical Corporation of India) in 120 ml of water. Then NaOH solution was mixed drop wise with regular interval of time with continuous stirring for 35 min. The mixture was allowed to stir for next 30 minute more and left it for another 48 hours. The obtained product was filtered using Whatman filter paper and washed properly with deionised water to remove all the sulphate ions etc. The white precipitate containing zinc hydroxide was spread on the glass substrate. The glass substrate was placed in an oven and alternate heating and cooling was applied. First of all it was heated at 150^0C for one hour then allowed to cool for 30 minute. The process of heating for one hour and subsequent cooling for 30minute was repeated for five times. Hence after total heating for five hour, ZnO-A is collected in powder form.

Similarly in method (B) again similar solution of NaOH and $ZnSO_4$ as in method (A) having same pH value and at room temperature were prepared. In this method NaOH solution was allowed to mix in one-shot in $ZnSO_4$ solution. The mixture was allowed to stir continuously for 12 hour then left for 72 hour. After 72 hour the mixture was filtered out by Whatman filter paper and washed properly through with deionised water to remove all the sulphate ions etc. After washing properly the residue i.e. zinc hydroxide was spread on a glass substrate. After applying similar process of heating and cooling as in method (A) i.e. subsequent heating for 1 hour and cooling for 30 minute for five times, ZnO-B is collected in powder form.

RESULT AND DISCUSSION

Surface morphology study by SEM:

The morphology of synthesized powder was investigated with scanning electron microscope (SEM, Edex). Figure 1(a) and (b) show the morphology of the ZnO film prepared by sol-gel technique for both methods. Method (A) and method (B) give the ZnO nanomaterial in the form of cluster and nanosheets showed in figure 1(a) and 1(b) respectively. The image obtained by SEM shows that the particle prepared by method (A) exist in cluster form whereas that obtained in method (B) does not exist in cluster form. The particles prepared by this process are named as ZnO- A and ZnO –B.

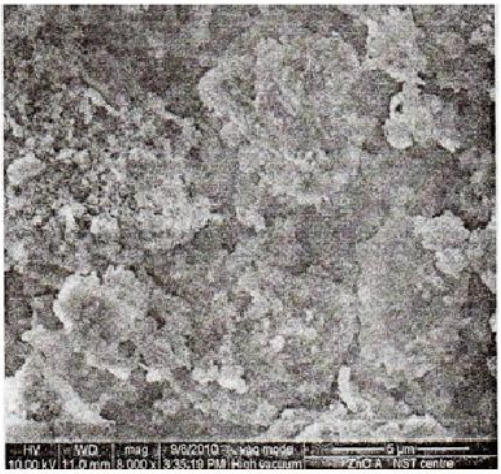

Figure 1(a): SEM image of ZnO - A prepared by method - A

Figure 1(b): SEM image of ZnO - B prepared by method - B

Crystal Structure study by XRD:

The crystalline property of synthesized powder was investigated with XRD (Mini Flex, Rinku Corporation, Japan). The X-ray diffraction data were recorded with Cu K_α radiation have wavelength 1.5 418 Å. The intensity data was collected over the range 10^0 to 70^0. The most intense peak (Fig.2 a and b) were considered for the sample ZnO- A and ZnO –B. the peaks are very intense which shows that the the grown material is crystalline in nature. The peak of ZnO –B is more intense with respect to ZnO- A. it shows that ZnO –B is more crystalline than ZnO- A. by calculating the FWHM values of the above sample, the sizes of the crystals are calculated by Debye Scherer Formula (Vermeulen*et al* 2007). The size of ZnO- A and ZnO –B were found 10nm and 7.7 nm respectively. The size of the particle prepared by the method as for ZnO –B is more fine with respect to ZnO- A.

The size of the crystal in the direction perpendicular to the reflecting plane is given by Debye Scherer Formula

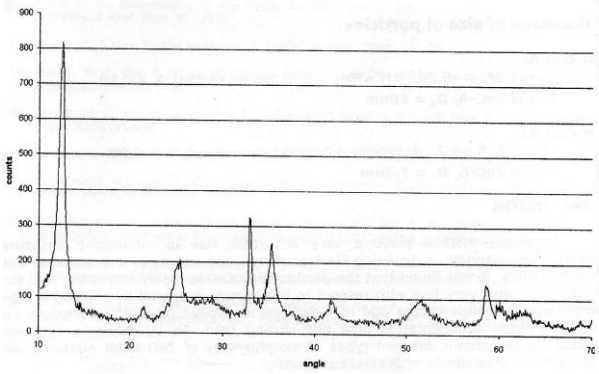

Figure 2(a) : XRD plot for ZnO-A

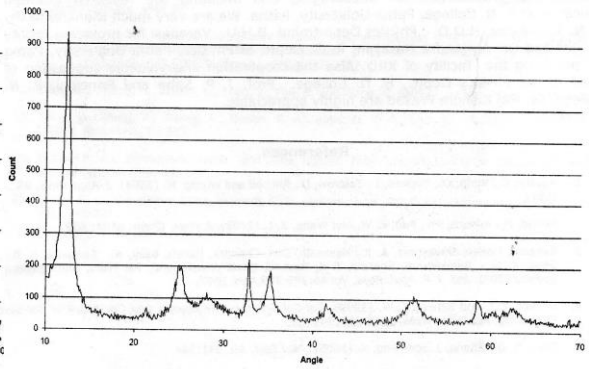

Figure 2(b): XRD plot for ZnO-B

$$D = \frac{k\lambda}{\beta cos\theta}$$

Where k is constant its value lies from 0.89 to1.39 depending on the specific geometry of the scattering object. For two dimensional lattices the value of k was found to be 0.89 while that of three dimensional lattices is 1.3. β is the FWHM of the peak in radians, θ is the diffraction peak position and λ is the wavelength of the x-rays used.

The experimental result was found to be

For ZnO- A: 2θ= 12.56, θ = 6.28, β (FWHM)=0.01396(in radian), K=0.89.
Size of ZnO-A, **D_1- 10.0nm**

For ZnO –B: 2θ= 12.40, θ = 6.20, β (FWHM)=0.01815(in radian), K=0.89.
Size of ZnO-B, D1- **7.7nm**

Conclusion

Synthesis method plays an important role in monitoring structural properties of materials as shown by the Scanning Electron Microscope and X-ray Diffraction study of ZnO nanomaterials. It was found that the particle of ZnO-B is highly crystalline and size of particle is also very fine with respect to the sample prepared by method –A. This morphological change due to difference in applied physical parameters i.e. due to different sedimentation time and stirring time. So, by changing synthesis method we can obtain different types of ZnO nanomaterials and change the crystallinity of ZnO nanomaterials.

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