



Hydrothermal Synthesis of Zinc Oxide – Aluminium Oxide Nanocomposite

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ABSTRACT

Composites are currently receiving much attention due to their tuneable chemical and physical properties. ZnO -Al₂O₃ composites attract particular interest because of their possible applications in dye-sensitized solar cells and sensors. Zinc oxide - Aluminum oxide composites were synthesized by hydrothermal method. The precursors used were Zinc Acetate, Aluminum nitrate and Sodium hydroxide. X-ray diffraction (XRD) pattern indicated that the synthesized Al₂O₃ was of hexagonal structure. Thermogravimetric analysis (TGA) was done to determine the thermal properties of the nanopowder UV-Vis spectroscopy and Dielectric analysis were done to determine the optical and dielectric properties of composite. The results were further discussed.

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Introduction

Zinc oxide (ZnO) is one of the II-VI oxide semiconductors with hexagonal wurtzite structure. It is an attractive candidate instead of GaN for short wavelength optoelectronic devices because of its wide band gap ($E_g = 3.37$ eV) and high exciton binding energy (60 MeV)[1]. In addition, ZnO can be extensively studied in surface acoustic wave devices, non-linear optical devices and photovoltaic equipment and so on [2].

There are many techniques to prepare nano particles, including Hydrothermal, Sol-gel, Solvothermal, Electro deposition and Chemical vapour deposition technique. Compared with the others, hydrothermal technique is simple, less expensive. In this paper, we report the synthesis of crystalline ZnO-Al₂O₃ composites by hydrothermal method. In addition, the effects of reaction temperature and reaction time on the properties of the ZnO-Al₂O₃ composites are also reported.

Experimental

In typical procedure an appropriate amount of zinc acetate and aluminum nitrate were taken separately and mixed in double distilled water which was stirred till the solutions became transparent. They were then mixed and stirred for 30 minutes. The as prepared solution was then transferred into a Teflon lined autoclave which was maintained between 180°C-230°C for 2 hours with a ramp temp of 1 hour. The autoclave was then cooled to room temperature and the obtain precipitates were washed several times with double distilled water and absolute ethanol to remove the impurities and then annealed 80°C for 15hrs.

The resulting hydrothermally synthesized ZnO/Al₂O₃ composites were subjected to different characterization including X-Ray diffraction (XRD), UV-Vis absorption spectroscopy (UV) and field emission scanning electron micrograph (FE-SEM). The crystalline structure of materials was analyzed by X-ray diffraction (RICH SIEFERT & CO with CuK α radiation $\lambda = 1.5406$ Å). The surface morphology was analyzed using Scanning electron micrograph (JEOL, JSM-67001). The absorption spectra were carried out in the range of

200 –1200nm by using CARY 5E UV-VIS-NIR SPECTROPHOTOMETER.

Results and discussion

Figure 1 shows the XRD pattern of the obtained products hydrothermally synthesized (180°C-230°C) for 2 hrs. The obtained product displayed the characteristic XRD peaks corresponding to hexagonal structure with lattice constants $a = 3.239$ Å and $c = 5.176$ Å (JCPDS tile no 89-7162) the average grain size calculated from X-Ray diffraction data by using Scherer's formula is 32.9nm

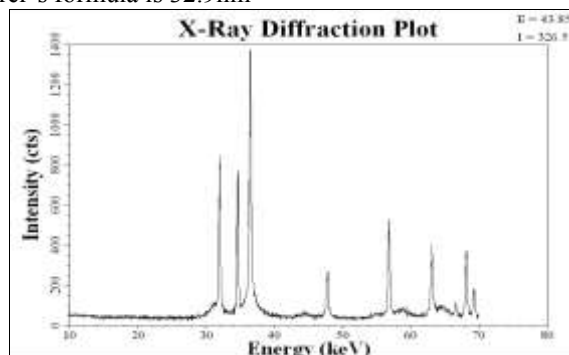


Figure 1 : XRD analysis of ZnO-Al₂O₃ composites

Figure 2 shows UV analysis of ZnO-Al₂O₃ composites annealed at 80° C for 15hrs. The absorption peak was observed at 371.87 nm. The band gap energy thereby calculated was found 3.347eV.

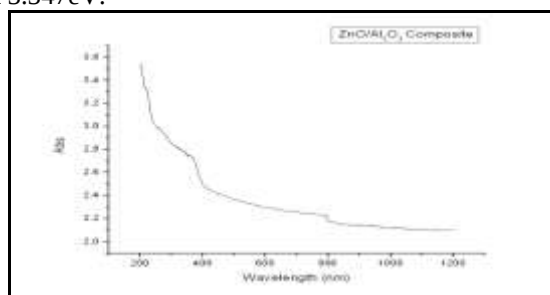


Figure 2 : UV-Vis Spectrum of the synthesized composite

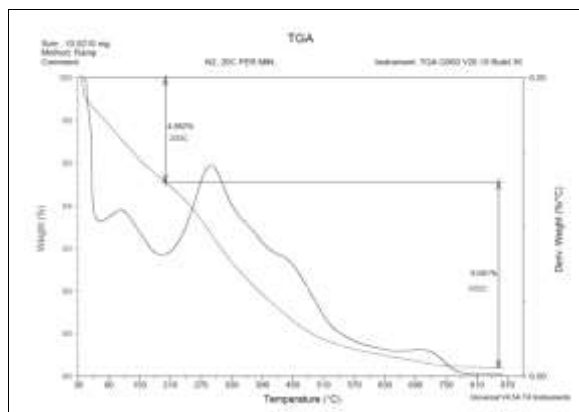


Figure 3 : Thermal Analysis of the ZnO/Al₂O₃ composite

The Thermal Analysis for the Zinc oxide /Aluminum oxide composites is given in figure 3. Zinc oxide and Aluminum oxide nanoparticles were heated from room temperature to 900°C in flowing Nitrogen. According to the TGA curve in the precursor shows a weight loss of 4.88% in the temperature range of 30 to 200°C and the next weight loss step at 850°C which is about 8.681%. The initial weight loss can be attributed to the loss of the remaining absorbed water and inter layer water. The peak around 330°C can be attributed to the combustion of organic materials and the start of nucleation. According to the curve, the mass loss increases with increasing temperature.

The surface morphology of ZnO/Al₂O₃ composite sample particles has been studied by Field emission scanning electron microscopy method. The images represent FESEM and EDAX images of the ZnO/Al₂O₃ mixed sample in figure 4. The FESEM image at a higher resolution reveals rod-like structure with around 200 nm length rods with width about 40 nm and around 30 nm small particles like structure found in the mixed sample.



Figure 4 : FE-SEM images of the as-synthesized ZnO/Al₂O₃ nanoparticles

The plot of the dielectric constant versus applied frequency is shown in figure 5. It is observed that the dielectric constant has high value in the low frequency region and thereafter decreases with the applied frequency. The high value of (ϵ_r) at low frequencies may be due to the presence of all the four polarizations namely space charge, orientation and, electronic and ionic polarization and the low values at higher frequencies may be due to the loss of significance of these polarizations gradually. This was observed in the ZnO/Al₂O₃ composite samples.

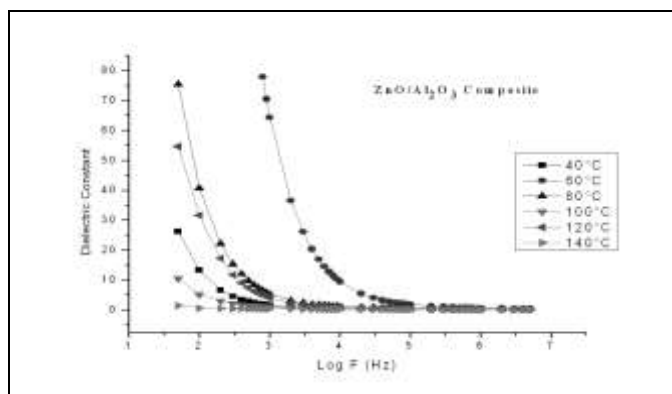


Figure 5 : Frequency vs Dielectric Constant for Zinc Oxide/Aluminum Oxide composites

Conclusion

Zinc Oxide as a wide band semiconductor is an extensively investigated material and Aluminum Oxide also known as alumina with its excellent dielectric properties and thermal properties makes it the material of choice for a wide range of applications. In recent years ZnO/Al₂O₃ composite has emerged as one of the most promising transparent conductive oxide. From the excitonic peaks of the UV absorption curve the band gap was calculated and found to be consistent with the direct band gap of the respective compounds.

The Thermal studies shows the nucleation of ZnO at 338.1°C, the formation of the metastable state in aluminum oxide and the weight loss percentages in the samples. FESEM provided the surface characteristics of the sample. The dielectric studies carried out on the samples helps us to understand the significance of dielectric properties of the samples respectively.

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