

Study Analysis of Photoconductivity of Zinc Oxide NPs and Bulk Zinc Oxide synthesized by Different Methods

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ABSTRACT

Here an attempt has been made to study photoconductivity properties of bulk ZnO and nano ZnO. Bulk ZnO synthesized by heat treatment and ZnO nanoparticles by sol-gel method at 500 °C temperature. Structural and morphological studies have been performed using XRD and SEM. XRD indicates that bulk ZnO is single phase with wurtzite structure and ZnO NPs (wurtzite structure) with crystallite size of the order of nm. SEM images of bulk ZnO indicate formation of spherical shape and SEM images of ZnO NPs show pseudo-spherical shape. UV-visible absorption study of synthesized ZnO NPs shows blue shifting of absorption edge as compared to that in bulk ZnO. Anomalous behaviour of photocurrent is observed in both samples wherein the photocurrent decreases even during steady illumination.

Keywords: XRD, SEM, Photoconductivity

1. INTRODUCTION

It is known that due to having specific properties and band gap energy of 3.37 eV [1-3] researcher has shown their great interest in Zinc Oxide. Due to Its band gap energy which lies in the ultraviolet (UV) range has generated a lot of interest in application in short wavelength optoelectronics, short wavelength light emitting diodes, laser diodes, solar cells, transistors and UV detectors [4-11].

Due to exhibiting different properties from bulk ZnO, nanostructures are more and more attractive and important. Properties like optical, electrical, semiconducting, piezoelectric, magnetic, sensing and transport effected due to high surface to volume ratio, surface defects and surface states in ZnO nanostructures. ZnO nanostructures play important role in their optical, electrical, semiconducting, piezoelectric, magnetic, sensing and transport properties. Hence so they are mostly used in gas sensors, photoconductors, photodiodes etc. [12-25]. Different types of zinc oxide nanostructures [26-34] may be form by different methods [35-41]. Some methods are complex while some are easy, some methods need long time while are short duration, some are costly and some are cheap. Among these methods, sol-gel method is facile, high yielding, suitable for preparing metal oxides, enabling mixing at an atomic level, thereby resulting in small particles making sintering easy.

There are various methods through which bulk zinc oxide (ZnO) can be synthesized such as oxidizing zinc metal directly, reducing zinc ore, or precipitating it from an aqueous solution. Bulk zinc oxide is having variety of application ranging from pigments and ceramics to sunscreens and antimicrobial agents. Among the various methods synthesizing methods of bulk zinc oxide heat treatment method is very simple and cheap. there are various methods to synthesize bulk zinc oxide such as.

Among the various properties of materials, photoconductivity property is very important property and has been studied. Photoconductivity property of ZnO nanostructures have been studied by several workers [42-48]. Photoconducting properties of nanoparticles depend on different parameters such as carrier density, process of carrier generation, presence of defect states, synthesis techniques, porosity, and adsorption and desorption processes and growth conditions etc. In addition, it also depends on applied voltage, intensity of light, wavelength of light and time. Zinc oxide nano structures are used in a variety of applications [49-58].

In a semiconductor photoconductivity is due to generation of electron-hole pairs in semiconducting material after absorption of a photon of suitable energy. In addition, there are also a number of studies on photoconductivity in nanoparticles, nanorods, nanowires of SnS, C, SnO₂, WO₃, FeH, CdS, ZnS, CdSe [59-68]. Due to wide band gap, high exciton binding energy and modified physical properties,

Similarly, Photoconductivity property of a large number of bulk materials photoconductivity properties of a large number of bulk materials such as ZnO, CdS, CdS-Se, CuInSe₂ ZnO/PVK, Graphene, TiO₂ ZnS, ZnO-CdO CdS-ZnO, TiO₂-ZnO. Some studies have been also done in form of single crystals, thin films and thick layers by several workers [59-65]

In the present chapter, ZnO NPs has been synthesized by sol-gel method at 500 °C and bulk ZnO has been synthesized by heat treatment method.

The synthesized samples have been characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), UV-visible absorption spectroscopy (UV-vis) and photoconductivity study (PC) in air. In the photoconducting properties by observing voltage dependence of dark current, rise and decay curve. Structural and morphological studies have been performed using XRD and SEM. UV-visible absorption spectra have been used to study optical properties.

2. EXPERIMENTAL SECTION

2.1 Chemical Section

For synthesis of Zinc oxide NPs, Zinc acetate (Zn(CH₃COOH)₂·2H₂O), Diethanolamine (DEA) and for bulk zinc oxide ZnO were purchased from E. Merk Ltd., Mumbai. The chemicals were directly used without any special treatment.

2.2 Sample Preparation

In 100 ml of double distilled water, 2.195 gm of zinc acetate was mixed and stirred for 8 hours. After 8 hours, a jelly was formed which was placed in furnace at 100 °C for 24 hours to get powder. The powder was further ground to get fine powder. The fine powder was placed in furnace at different temperatures (400, 500, 600, 700 and 800 °C) for 4 hours to synthesize five different samples of ZnO NPs. All the samples were further ground to be used for cell preparation.

The samples were prepared with heat treatment technique. Pure form of ZnO powder was taken and filled in ceramic boat and fired in muffle furnace with controlled air atmosphere. Then, it is cooled down to the room temperature and ground in a mortar to get microcrystalline form of the powder of ZnO. Sample was fired at 500°C for 30 min.

2.3 Instrumentation

The crystal structure of ZnO nanoparticles and bulk ZnO was characterized by X-Ray diffraction using Rigaku D/MAX-2200H/PC with Cu K α radiation ($\lambda = 1.54178 \text{ \AA}$). For investigating photoconductivity and dark conductivity, a cell is formed by spreading a thick layer of powdered sample in between two Cu electrodes etched on a Cu plate (PCB), having a spacing of 1 mm. The powdered layer was pressed with transparent glass plate. This glass plate has slit for providing illumination area of 0.25 cm². In this cell type device, the direction of illumination is normal to the field across the electrodes. The cell was mounted in a dark chamber with a slit wherefrom the light is allowed to fall over the cell. The visible photo response was measured using of 300 W mercury lamp as photo excitation source. A stabilized dc field (50 V/cm to 500 V/cm) was applied across the cell to which a digital dc nano meter for measurement of current and Rish multi 18S with adapter were connected in series. The cell is kept in dark till it attains equilibrium before measuring the photoconductivity.

3. RESULTS AND DISCUSSION

3.1 Structural study

Figure 3.1 shows XRD patterns of nano zinc oxide nano particles and in inset bulk zinc oxide. ZnO nano particles (NPs) synthesized by sol-gel method and bulk ZnO synthesized by heat treatment method at room temperature.

The X-ray diffraction patterns of bulk ZnO (in inset) indicates that ZnO is single phase with wurtzite structure and for ZnO NPs synthesized at different temperatures the peaks are observed at (100), (002), (101), (102) and (110). All peak positions and relative peak intensities for all the samples belong to a wurtzite structure corresponding to JCPDS card no.75-0576 [66].

In bulk ZnO it can be seen that the diffraction peaks are higher and narrower, implying that ZnO is well crystallized. The peak corresponding to lattice plane (101) is most prominent and no others peaks of impurities are observed which indicates that the prepared ZnO is of high purity. Average crystallite size is calculated using Scherer formula [67].

$$D = \frac{0.9\lambda}{\beta \cos \theta} \quad (3.1)$$

where D is the crystallite size, λ is the X-ray wavelength used in XRD, θ is the Bragg angle and β is the full-width at half-maximum (FWHM) measured in radian of the concerned peak. The crystallite size calculated for most prominent peak of ZnO synthesized at different temperatures of 400, 500, 600, 700 and 800 °C is 23.12 nm, 29.56 nm, 38.45 nm, 58.32 nm and 76.32 nm respectively i.e. average crystallite size lie in the range of 23 nm to 76 nm.

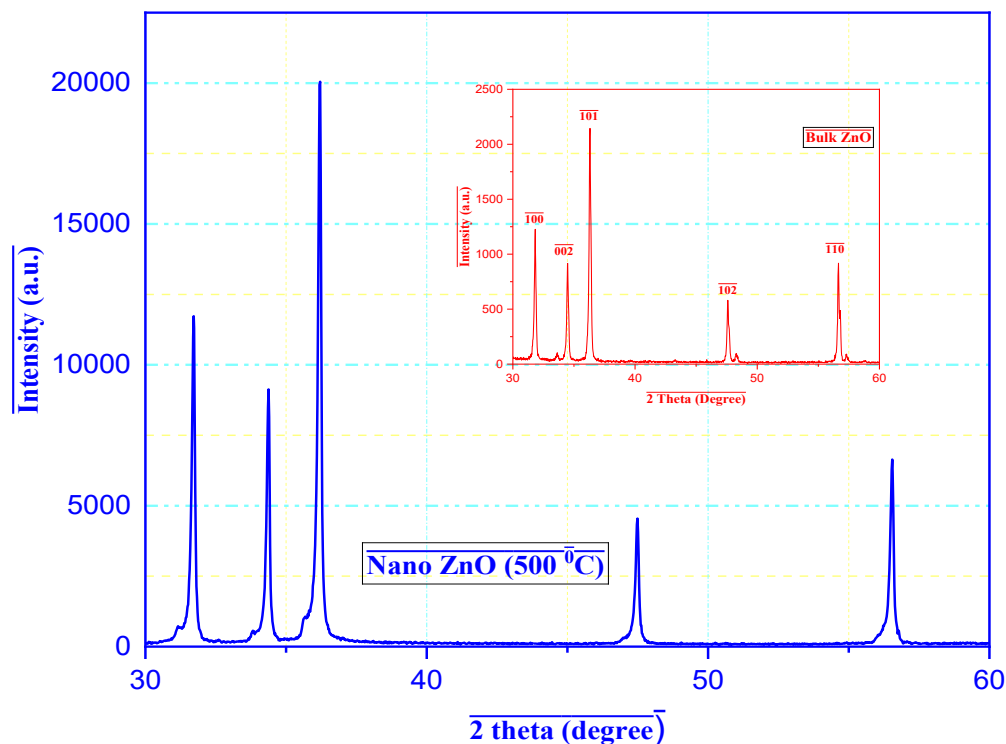


Fig. 3.1 X.R.D of Zinc Oxide NPs (by Sol-gel method) and in inset Bulk Zinc Oxide (by Heat treatment method)

3.2 Morphological study

Figure 3.2(a) shows SEM image of ZnO NPs synthesized at 500 °C. As it is clear from the figure that the morphology of particles is pseudo-spherical and its grain size is near about in the range of nanometer. The nanoparticles are seen merging with each other and forming a neck between two particles. Such a neck formation may lead to densification of the particles [68-69]. Figure 3.2 (b) shows SEM image of bulk ZnO synthesized by heat treatment method. The particles shape of ZnO are having spherical shapes and size calculated from the SEM image is found to lie in the range of 300-500 nm.

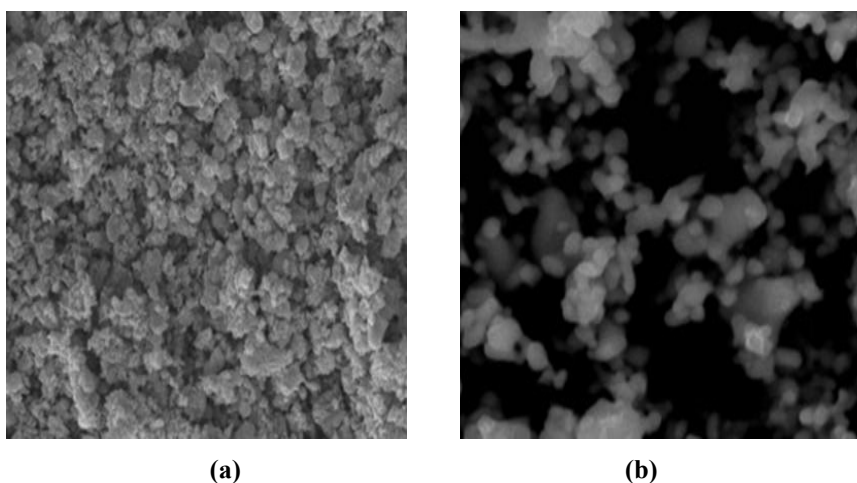


Figure 3.2 SEM images of ZnO NPs synthesized at (a) 500 °C (b) Bulk ZnO

Figure 3.3 shows TEM micrographs of ZnO nanoparticles synthesized at 500 °C. The morphologies of all nanoparticles are found to be nearly spherical in nature with the diameters ranging from 30 to 50 nm. The estimated values, obtained using TEM micrographs, are in close agreement with those obtained from XRD data. The particle size observed in TEM

measurement is more than the crystallite size that indicates agglomeration of crystallites in ZnO nanoparticles.

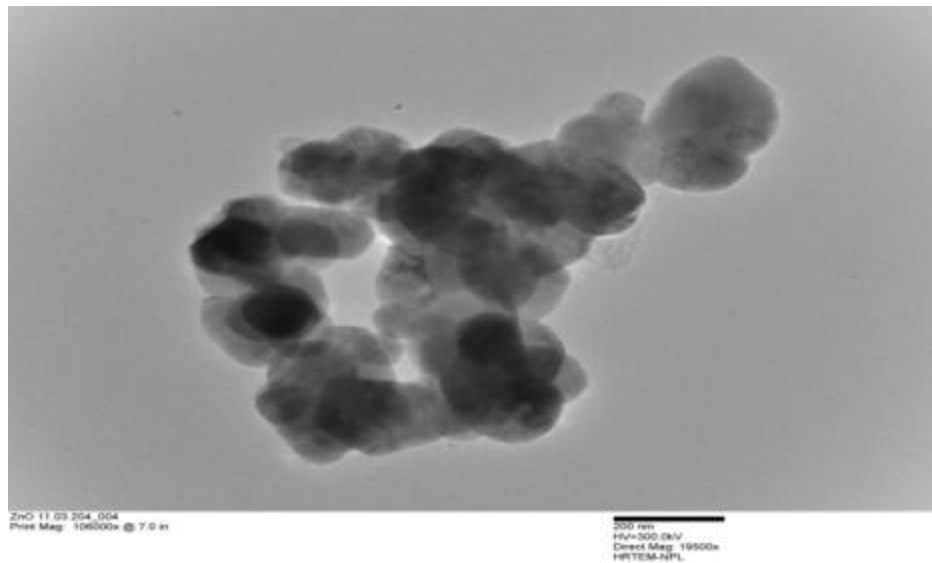


Figure 3.3 TEM images of ZnO synthesized at 500 °C.

3.3 UV-visible absorption

Figure 3.4 shows the UV-visible absorption spectrum of bulk ZnO and ZnO NPs. As it clear in the Figure 3.4 (a) that the occurrence of a small peak in bulk ZnO shows that the absorption edge appears at 380 nm which is higher than the absorption edge of bulk ZnO [70]. The red shift may be attributed to change in lattice structure/ parameter. Figure 3.4 (b) shows UV-visible absorption spectra of ZnO NPs synthesized at 500 °C. The absorption edge is blue shifted as compared to that in bulk ZnO [71-73] which may be attributed to nano dimension of ZnO particles. XRD results.

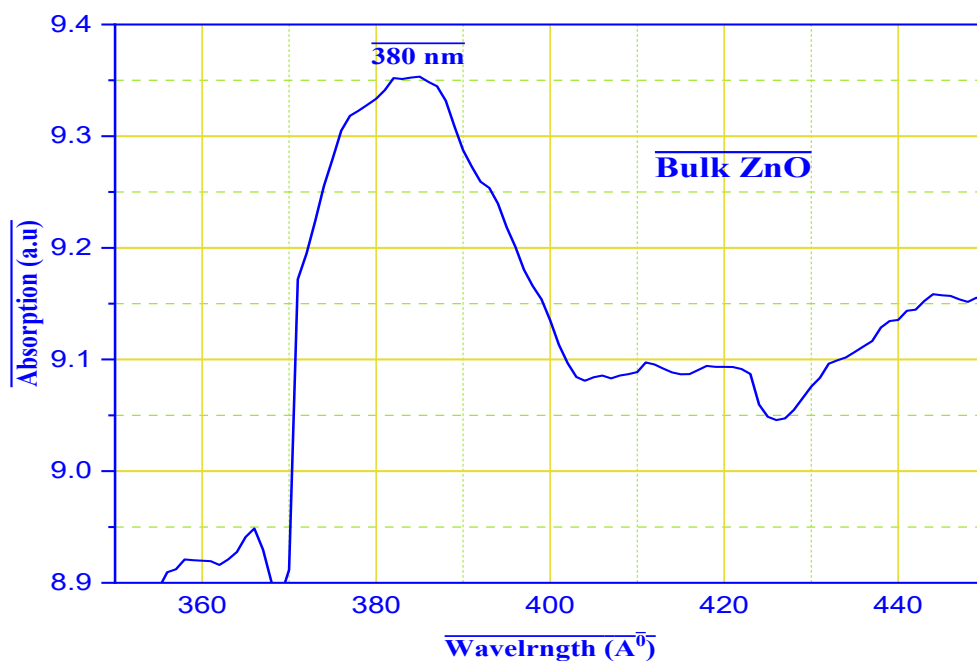


Figure 3.4 (a) UV visible absorption spectrum of bulk ZnO

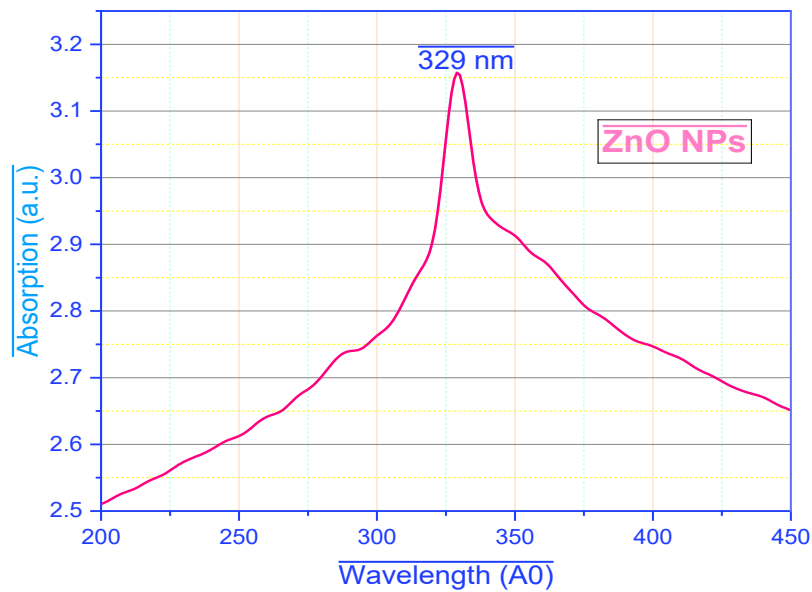


Figure 3.4 (b): UV visible absorption spectrum of Nano ZnO

3.4 Photoconductivity study

3.4.1 Voltage dependence of dark current

Figure 3.5 shows variation of dark current (I_{dc}) with applied voltage (V) on \ln - \ln scale for bulk and Nano ZnO. $\ln I_{dc}$ versus $\ln V$ curves are straight lines having different slopes and can be represented by $I_{dc} \propto V^r$, where ' r ' is the slope of different line segments. At low voltage, bulk ZnO as well as ZnO nanoparticles are found to exhibit super linear dependence of dark current on applied voltage which may be attributed to injection of additional charge carriers from one of the electrodes [74-77]. At high voltage, variation of dark current with applied voltage remains super linear for both bulk ZnO and as well as ZnO NPs all samples but ZnO is found to vary with power indices greater than 2. This may be attributed to space charge due to excess carriers injected from electrodes with respect to steady state carrier concentration [74-76].

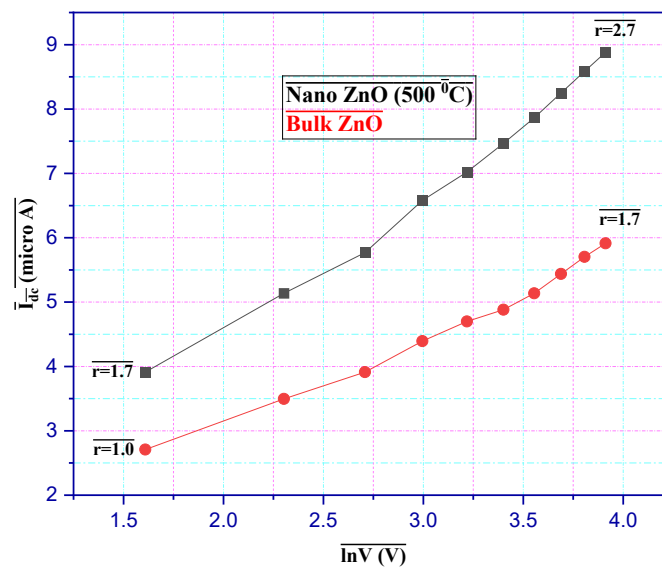


Figure 3.5 Variation of dark current (I_{dc}) with applied voltage (V)

3.5 Rise and decay of photocurrent in air

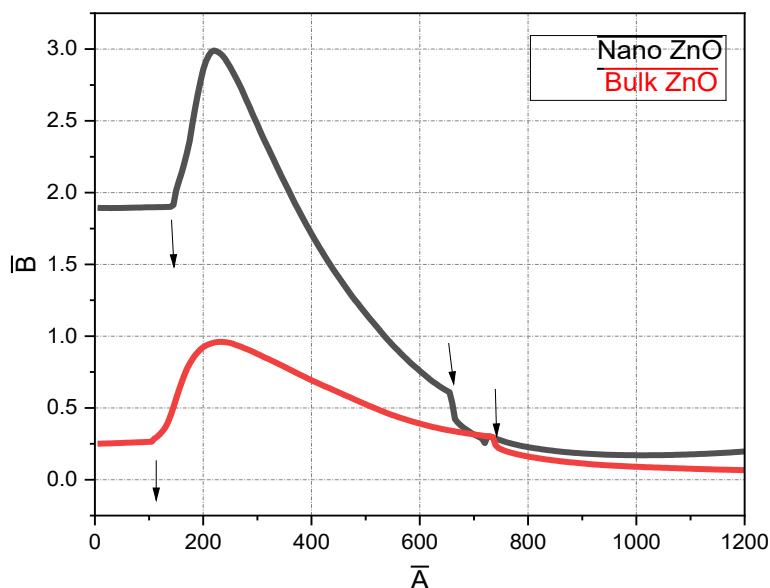


Figure 3.6 Rise and decay curves for bulk ZnO and ZnO NPs in air

Figure 3.6 shows time resolved rise and decay of photocurrent for ZnO NPs synthesized at 500 °C temperature and bulk ZnO with bias voltage of 20 V at room temperature. The cell is first kept in dark till dark current for the both samples becomes stable. For the both samples, as the light is switched on, the photocurrent increases very fast initially and attains a peak value. The reason behind initial sharp increase in photocurrent is because of fast process of generation of carriers as a result of absorption of photons. Further, it starts decreasing. Eventually, it gets stabilized. Negative photoconductivity is defined as the decrease of conductivity in the presence of light. The negative photoconductivity is due to presence of imperfection centres in the forbidden gap [78-79]. When illumination of the sample increases the density of the electrons or holes or both, positive photoconductivity results. If with the elapse of time, the minority carriers are also excited from the imperfection centres, negative photoconductivity results. This is due to recombination of minority carriers with majority carriers. All the samples are found to exhibit negative photoconductivity. This effect is more prominent in pure ZnO. When the light is switched off for the both samples, the photocurrent initially decreases exponentially due to recombination of photo generated carriers and further it gets stabilized at a value lower than dark current for the samples. In the both samples resulting in negative photoconductivity (NPC). Similar anomalous photoconductivity behaviour has been reported by many authors [80-84]

4. CONCLUSIONS

In the present work, bulk ZnO are synthesized by heat treatment method and ZnO NPs have been synthesized by sol-gel method at 500 °C temperature. Particle size calculated from XRD and SEM are found to lie in the range of 200-550 nm for bulk ZnO and ZnO NPs lies in the range of 23-76 nm. Formation of pseudo spherical ZnO nanoparticles can be seen by SEM images. Blue shifting of absorption edge is exhibited by UV-vis absorption spectra of ZnO NPs. For the bulk and nano ZnO variation of dark current with applied voltage exhibits super linear behaviour. In the both samples photocurrent decreases even during steady illumination.

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