

## ZnO NEEDLE-LIKE STRUCTURES: SYNTHESIS AND CHARACTERIZATION

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### ABSTRACT

We report in this paper, the structural and I-V properties of ZnO nano needle-like structure synthesized by Co-precipitation method. X-ray diffraction (XRD) result shows that the ZnO nano needle-like structure with hexagonal phase and no secondary phase was observed. The crystallite size has been calculated by Scherrer's equation which was found to be in the range 40-60 nm. SEM images reveal that ZnO nano needle-like structure has the length of ~5.5  $\mu\text{m}$  and base of ~5  $\mu\text{m}$  are consistent with the results from SEM investigations. I-V characteristics have been carried out to study the conducting behaviour of the prepared ZnO nano needle-like structures.

**Keywords:** ZnO nano needle-like structure, Co-precipitation, I-V characteristics, SEM.

### 1. INTRODUCTION

Nanoscale semiconductor materials have attracted great interests of researchers because of their importance not only in fundamental research areas but also in practical applications. ZnO nanostructures have been studied intensively and extensively over the last decade not only for their remarkable chemical and physical properties, but also for their current and future diverse technological applications. ZnO is a typical inorganic semiconductor and piezoelectric material; this material has a direct wide band gap of 3.37 eV and a large exciton binding energy of 60 meV at room temperature (Ozgur *et al.*, 2005). It has enormous applications in electronic and electromechanical devices (Wang, 2009), such as ultraviolet (UV) lasers (Kota *et al.*, 2011), high performance nanosensors (Zhou *et al.*, 2009), solar cells (Weintraub *et al.*, 2009), piezoelectric nano generators (Yang *et al.*, 2009), and nanopiezotronics (Wang, 2008). In order to grow one-dimensional (1D) ZnO nanostructures, various techniques have been developed like wet chemical methods (Narkiewicz *et al.*, 2008), physical vapour deposition (Onur *et al.*, 2011), pulsed laser deposition (Hong *et al.*, 2009), sputtering (Dang, 2007). This article gives a comprehensive overview of the progress that has been made within the context of one-dimensional (1D) ZnO nanostructures synthesized via chemical precipitation method.

### 2. EXPERIMENTAL

ZnO nano needle have been prepared using the required precursors by chemical precipitation method. An aqueous solution of 1M Zinc acetate dihydrate  $\{\text{Zn}(\text{CH}_3(\text{COO}))_2 \cdot 2 \text{H}_2\text{O}\}$  dissolved in water and stirred for about 30 min at room temperature. Sodium hydroxide (NaOH) (0.7 M) was added drop wise to the above mentioned solution. The colour of the solution changed into milk white coloured, indicating the formation of ZnO nano particles in the solution. The solution was stirred for 10h at room temperature. After 10h the supernatants were removed and the deposited precipitate was centrifuged and washed with water and ethanol several times. The samples were then suspended in ethanol and allowed to age for 6h without stirring. After centrifugation, the samples were then dried in oven at 60°C for 2 h. Then, the as prepared ZnO nanoparticle is placed in the middle of a muffle furnace in silica crucible. The samples have been annealed at 400°C for one hour.

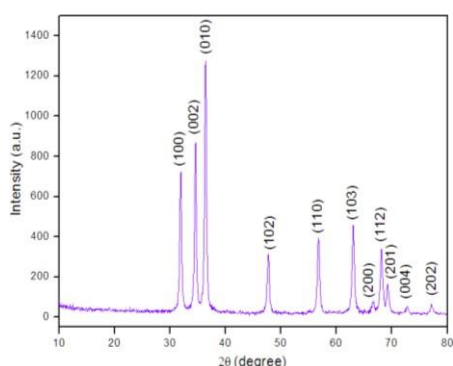
#### 2.1 Characterization of ZnO nanoparticles

X-ray diffraction studies have been carried out using PANalytical x-ray diffractometer and surface morphology and the compositional analysis of the samples has been studied using scanning electron microscope (JEOL JSMS 800-V) and energy dispersive analysis studied using the prepared ZnO nano needle-like structured samples have been recorded using a JEOL JEM2100 microscope. I-V characteristics of ZnO nano needle-like structured

samples have been recorded by using four-probe method.

### 3. RESULTS AND DISCUSSION

Fig. 1 shows the X-ray diffraction patterns of the ZnO nano needle-like structure. The diffraction peaks at  $2\theta$  (degrees) of  $31.63^\circ$ ,  $34.61^\circ$ ,  $36.32^\circ$ ,  $47.66^\circ$ ,  $56.94^\circ$ ,  $62.97^\circ$ ,  $66.57^\circ$ ,  $68.12^\circ$ ,  $69.48^\circ$ ,  $72.11^\circ$  and  $72.26^\circ$  are respectively indexed as the (100), (002), (101), (102), (110), (103), (200), (112), (201), (004) and (202) planes of ZnO. All the diffraction peaks in the  $2\theta$  range measured corresponds to the hexagonal structure of ZnO with lattice constants  $a = 3.253\text{\AA}$  and  $c = 5.214\text{\AA}$  and are in good agreement with those on the standard data card (JCPDS card No. 36-1451).



**Fig. 1. X-ray diffraction pattern of as prepared ZnO nano needle-like structure**

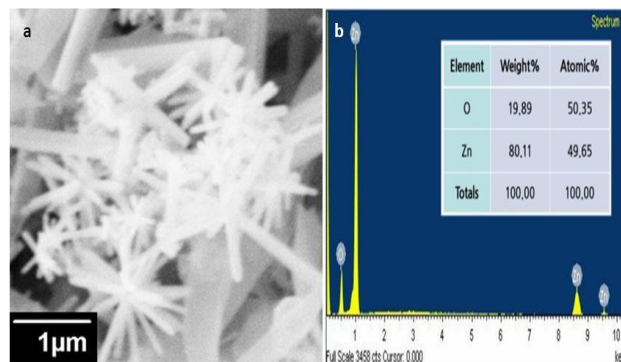
The sharpness of the diffraction peaks suggests that the product is well crystallized. The crystallite size of ZnO is calculated using Scherrer's equation

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

where,  $D$  is the crystallite size,  $K$  is a constant taken to be 0.94,  $\lambda$  is the wavelength of the X-ray radiation,  $\beta$  is the full width at half maximum and  $\theta$  is the angle of diffraction. The crystallite size has been calculated and is found to be in the range 40-60 nm for as prepared ZnO nano needle-like structure.

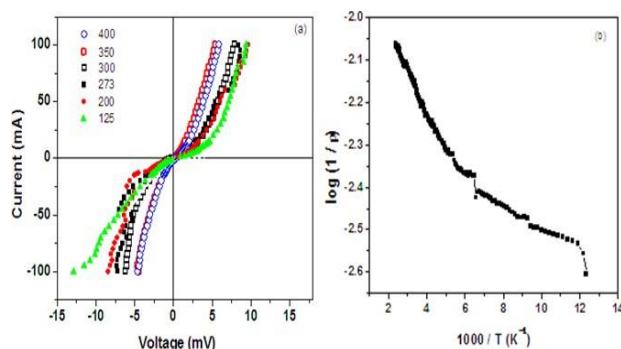
Fig. 2a displays an SEM image of the sample prepared with zinc acetate and sodium hydroxide as reactants under conventional conditions, from which it can be seen that there are many nanorods with flat ends, are seen to arise from centre, it gives the appearance of a needle, their average diameter are 20 to 30 nanometers and length varies from 70 nanometers to  $5\mu\text{m}$  and the average size of whole needle is  $5\mu\text{m}$  shows needle-like structures. The size of the complex structure and the diameter and

length of the ZnO nano needle-like structure are consistent with the results from SEM investigations. Energy dispersive X-ray analysis (EDS) of ZnO nano needle-like structures are shown in Fig. 2b. The chemical constituents present in the ZnO sample are of Zn-49.65% and O-50.35%. In the EDS, Zn and O are the element detected, indicating that the sample is highly pure.



**Fig. 2. a) SEM images of ZnO nanoparticles and b) EDS spectra of ZnO nanoparticles**

For I-V measurements in bulk, pellets of 13 mm diameter and thickness  $\approx 1$  mm were prepared under a load of 5 tons. These pellets were used in Four-Probe method. The I-V characteristics of the samples were studied at the room temperature as well as at various temperatures (125K, 200K, 273K, 300K, 350K and 400K) using the Four-Probe method. The temperature dependence of resistivity was measured at constant current by varying the temperature continuously. The silver paste was used for Ohmic contact between the sample and the copper probes. DC voltage across the electrodes was measured by varying the current. I-V plots are shown in Fig. 3(a) which shows the temperature dependence of I-V characteristic of ZnO nano needle-like structure as a representative case.



**Fig. 4 (a) I-V characteristics (b) Conductivity Vs Temperature ZnO nano needle-like structure sample**

I-V characteristics of the samples are measured in presence of argon gas at low as well as at high temperatures. Liquid Nitrogen is used for lowering the temperature. Argon gas is necessary to eliminate the moisture content otherwise the moisture present in air will change the electrical properties of the sample (especially at low temperatures). Fig. 3(a) depicts the characteristics of pure ZnO at the constant current, where the voltage decreases as we go on lowering the temperature. The conductivity of the sample increases with increasing temperature, as shown in the Fig. 3(b) and in the Table 1.

**Table 1. Temperature with Conductivity**

T(K)	$\sigma(\Omega^{-1})$	R( $\Omega$ )
400	0.66511	1.5035
350	0.77082	1.2973
300	0.85095	1.1751
273	1.08472	0.9219
200	1.25094	0.7994
125	1.66917	0.5991

#### 4. CONCLUSION

The structural and electrical properties of ZnO nano needle-like structure was synthesized by a simple chemical bath deposition method. The sizes of the needle-like structures are about  $\sim 5.5\mu\text{m}$  on an average. The length and breadths of the needle-like structure are about  $\sim 5.5\mu\text{m}$  and  $\sim 5\mu\text{m}$ . The size of the complex structure and the length and breadths of the ZnO nano needle-like structure are consistent with the results from SEM investigations. The conductance of the ZnO nano needle-like structure was estimated from the I-V characteristic from lower temperature to higher temperature. The conductance of the sample increases with decreasing temperature. The increase in the conductivity can be attributed to the increase in the electrical property.

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