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Electrical Characterization of ZnO Nano Composites†

ARNAB GANGOPADHYAY*, ADITI SARKAR and A. SARKAR

Department of Physics, Bijay Krishna Girls' College, 5/3 M.G. Road, Howrah-711101, India

*Corresponding author: E-mail: agangulyphysics@gmail.com

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In this work ZnO nano clusters were grown in biopolymer, gum acacia background. Attempt has made to control the nano-cluster size by *in situ* chemical sol-gel process through the control of the rate of forward reaction. X-Ray diffraction study and optical absorption study and D.C. conductivity study on the nano clustered material were done to get information on grain size. An empirical formula relating nano-size and conductivity fluctuation has been proposed here on the basis of preliminary observation. The work indicates a possible new characterization technique by the use of the D.C. I-V characteristics. Results of electrical transport through the developed nano-composites are found to exhibit dominant quantum effect at room temperature.

Key Words: Nano-composites, Spectrophotometry, Electrical conduction, Optical absorption.

INTRODUCTION

Nano-composites occupied a leading role in the field of science, technology and material science. Organic-inorganic nano-composites are of considerable interest and classified into two types: nano-particles and nano-structured materials. Metal oxide nano-composites exhibit a variety of electronic[1], magnetic and structural material properties. Due to extremely small size the surface to volume ratio is very high. That is why nano-composite's show high chemical activity. Zinc oxide finds various applications in medical science because it does not show adverse chemical effect in human body. Zinc oxide nano-composite's may be applied in medical application for drug delivery or opto-chemical applications. The material is important in development of solar cells due to their small cluster size (~ 50 nm). Generally preparation of nano-composite is achieved through top-down (ball-milling, X-ray lithography *etc*.) or bottom-up process. In this work bottom-up sol-gel route in relatively low temperature (~100 ºC) is followed. The process uses zinc acetate as main reagent and gum acacia as a capping agent. It is important to study the properties and cluster size of a nano-composite sample for better application. The objective of the present work aiming at a new characterization technique from the study of D.C. electrical character is legitimate as a quick, easy and cost effective method compared to other micrographic techniques.

EXPERIMENTAL

In situ production of ZnO nano-composite was prepared from zinc acetate, analytical grade (CDH, India) and gum acacia (E. Merck Ltd., India) as starting materials. The later is a high potential biopolymer[2]. 2.20 g of $Zn(CH_3COO)_2$ was dissolved in each 10, 20, 40 and 50 mL distilled water (sample A, B, C, D respectively). About 30 g of gum acacia powder was dissolved in 200 mL hot distilled water. Then each $Zn(CH_3COO)_2$ solution was mixed in 50 mL gum acacia solution drop wise at boiling temperature (~104 ºC) (sample A,B,C and D respectively). The refluxing was continued for about 5 h. After completing the reaction, final product was caste to form measurable pellets on adequate drying.

Detection method: DC volt-ampere (I-V) characteristics of the developed nano-composites were measured at room temperature with 20 mV voltage steps and 40 mV steps (0 to 2V) using PC interfaced Keithley 2400 (USA) source meter to have information on charge transfer. Optical absorbance of first sample was measured with Systronics 2201 (India) UV-VIS spectrophotometer to get the bandgap of the developed nano-size particles. XRD of ZnO specimens were measured with Rigaku Miniflex, Japan using CuK_α line to get the grain size.

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RESULTS AND DISCUSSION

It has been found that ZnO nano-composite can be developed through bottom up sol-gel process[1]. Fig. 1(I) shows the XRD pattern of developed ZnO nano-composite (sample A) and compared with ordinary ZnO powder [Fig. 1(II)] with a clear distinction.

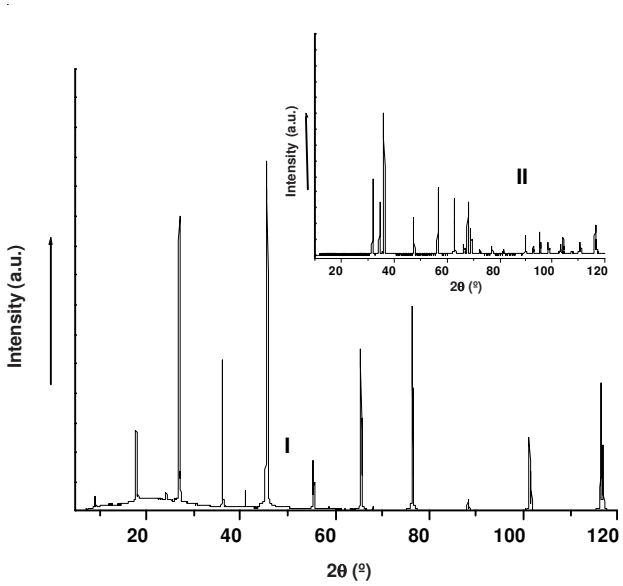

Fig. 1. XRD pattern of sample A and bulk ZnO (inset)

Using Scherrer formula[3], the estimated average grain size of the ordinary ZnO is found to be 98.39 nm whereas that for developed ZnO nano-composite has the average grain size 42.22 nm. D.C. conductivity estimated from measured I-V characteristics data *vs.* applied voltage for the mentioned specimen are shown in Fig. 2 (sample A and B) and Fig. 3 (sample C and D), respectively. It shows significant differences for two types of nano-composite those are produced under different reaction condition.

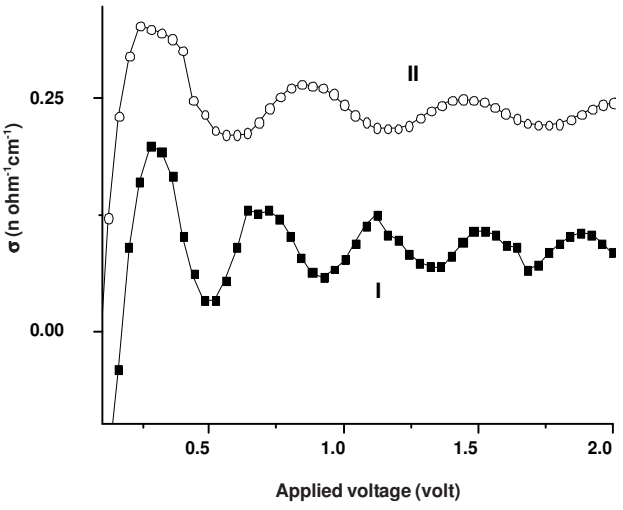

Fig. 2. Conductivity *vs.* applied voltage curve for sample A (I) and sample B (II)

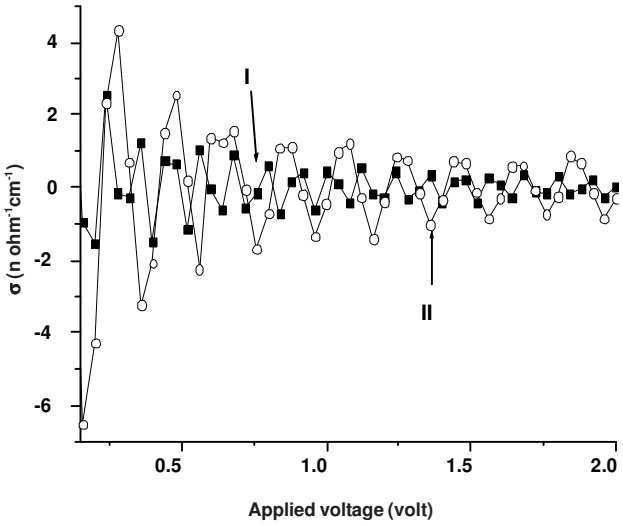

Fig. 3. Conductivity *vs.* applied voltage curve for sample C and D (I and II, respectively)

It is also established that the slope of D.C. characteristics are different for different nano-composites[1]. The sol-gel reaction was driven in four different reagent [$Zn(CH_3COO)_2$] concentration. Since this process is essentially a bottom-up process, highly active nascent ZnO molecules are first coagulate to produce relatively inert larger clusters and those are encapsulated by biopolymer surfactant gum acacia. The coagulation rate and capping mechanism depend on different reagent concentration which in turn provides a way of controlling cluster size of developed nano-composite's. The conductivity plots reflect this fact. The close similarity of ZnO bulk conductivity plot in Fig. 4 for 20 and 40 mV step size implies that the result is not due to 1/f noise. In Fig. 2 conductivity of sample A and sample B is depicted and in Fig. 3 conductivity of sample C and sample D is depicted, all measured in 40 mV step size. Table-1 summarizes the observation on conductivity characters of Figs. 2-4 (graph II).

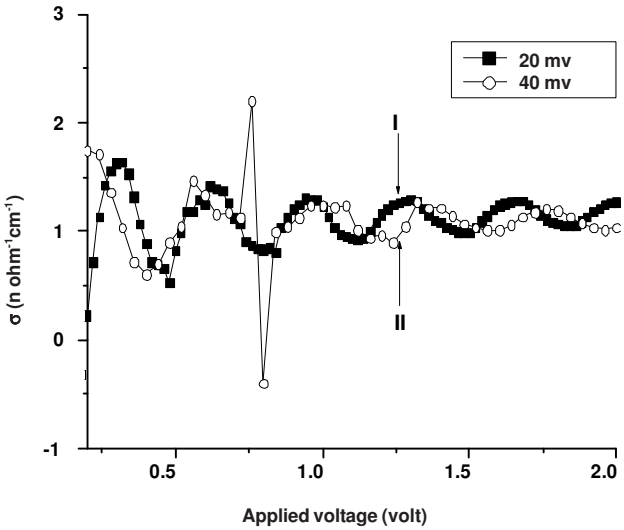

Fig. 4. Conductivity *vs.* applied voltage curve for bulk ZnO in 20 mV and 40 mV step (I and II, respectively)

On this primary observation we can propose a simple empirical formula which can approximately encompasses the result:

TABLE-1
AVERAGE PEAK WIDTH IN CONDUCTIVITY *vs.* APPLIED VOLTAGE CURVE

Sample	V_1	V_2	N	$\Delta V = \frac{(V_2 - V_1)}{n}$
A	0.68	1.52	2	0.42
B	0.84	1.48	1	0.64
C	1.12	1.36	2	0.12
D	1.08	1.44	2	0.18
Bulk sample	0.96	1.66	2	0.35

Here V_1 and V_2 are peak position in conductivity curve and n is the number of maxima peaks of conductivity curve between V_1 and V_2.

$$d.\Delta V = \text{constant} \quad (1)$$

where d is the grain size and ΔV is the voltage difference for two consecutive maxima of conductivity at V_1 and V_2.

Fig. 5 shows the optical absorption spectra of ZnO nano composites, which shows a absorbance peak at 381.5 nm corresponding to 3.26 eV. ZnO has bulk band gap 3.2 eV[4] corresponding to 388.4 nm wavelength. This corresponds to blue shifted band gap 3.26 eV due to quantum size effect which also indicates the presence of nanosized ZnO particles[5,6].

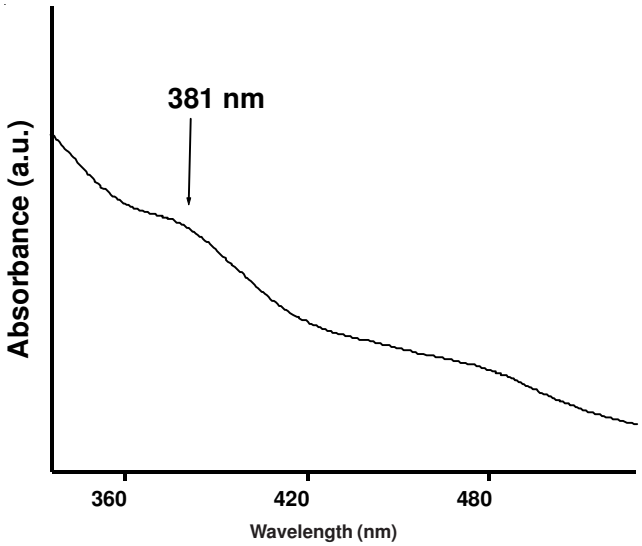

Fig. 5. Optical absorption spectrum for ZnO nano composites

Conclusion

Gum acacia capped ZnO nano-composite have been prepared successfully by chemical sol-gel route. The D.C. conductivity curves as function of applied voltage for developed ZnO nano-composite shows definite changes as sample containing different particle size changes. From the behaviour of the conductivity fluctuation curve an empirical relationship between grain size and average peak width of conductivity curve has been proposed. This provides a new characterization method for estimating grain size of nano-composite.

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