ISSN 0974 - 9373

Vol. 14 No.2 (2010) **Journal of International Academy of Physical Sciences** pp. 245-250

Synthesis, Dielectric and Electrical Characterization of SnO_2 Nano-particle Prepared by Co-precipitation Method*

Raman Mishra and P. K. Bajpai
Advance Material Research Laboratory
Department of Pure & Applied Physics
Guru Ghasidas Vishwavidyalaya, Bilaspur 495 009
E-mail: bajpai.pk1@gmail.com

(Received February 20, 2010)

Abstract: Nano-crystalline SnO_2 particles have been synthesized by co-precipitation method using a simple starting solution consisting of $SnCl_2.2H_2O$ and NH_4OH as complexing and ethanol as polymerization agents. The structural properties of the prepared tin oxide nano-powders annealed at different temperatures (200^0C–700^0C) have been characterized by X-ray diffraction (XRD).The XRD pattern shows tetragonal phase in the nano powder with lattice parameter a=4.7383A0 c= 3.1948 A^0 .The Size of the prepared material is estimated to be ≈23 nm crystals. Impedance spectroscopy measurements taken in the temperature range between RT and 150 ^0C show a clear dependence of the measured impedance on the particle size. The impedance analysis reveals grain resistance decreasing with temperature as expected for a semi conducting material.

Keywords: Semiconductor nano-particles, co-precipitation method, X-ray diffraction, Impedance analysis.

1. Introduction

Tin oxide (SnO_2), with cassiterite structure is a wide band gap n-type semiconductor (E_g =3.6–3.8 eV), and one of the most widely used semi-conducting oxides due to its chemical and mechanical stabilities. It has been widely studied over decades because of its applications in various solid state and ceramic devices, sensors, optoelectronics and catalysis[1-6]. Among the technical applications, the most important uses of SnO_2 are as gas sensors. The sensing properties of SnO_2 sensors (sensitivity, selectivity and reproductively) critically depend on particle size and specific surface area. Therefore, preparation of primary powders as nano-particles increases specific surface area of the particles and hence improves the efficiency function of the sensor[1, 5-7]. Due to these modifications application of nano-structured SnO_2 as an active material in gas sensor is well known. Recently,

*Presented at CONIAPS XI, University of Allahabad, Feb. 20-22, 2010.

polymerizing–complexing (PC) combustion method, without any precipitation, has been used for preparation of semi conducting nanoparticles. This method consists of a variety of organic fuels such as citric acid, urea, hydrazine, EDTA and polyethylene glycol as complexing or polymerization agent[7]. PC method provides more homogeneous fine powder than any other techniques with immobilization of metal–chelate complexes and forming stable metal complexes via increasing the polymerization. The main purpose of the present research is to study the post-annealing temperature effect on nano-particle size and characterize its electrical properties.

2. Experimental

SnO_2 nano- powder is synthesized by the Co-Precipitation method. 0.5 gm of $SnCl_2.2H_2O$ was treated with 25% NH_4OH solution added drop wise with maintaining pH till 8. The white precipitate thus obtained was washed successively with deionized water and ethanol to remove any unreacted salt and excess ammonia. This washed precipitated was then dissolved in certain amount of ethanol and was peptized. After rigorous stirring at 60^oC for 30 minutes white precipitate left. Finally wet white precipitate was fully dried on hot plate at 80^0C for 30 minutes resulting into dry powder. The precursor powder, which has been prepared was annealed at 200^oC, 400 ^oC, 700 ^oC for 2 hrs in glassy boat in an electric box furnace and then cooled down to room temperature.

3. Result and Discussion

Mass loss during annealing from 200^0C to 500^0C occurs due to evaporation and removal of organic additives. The most mass reduction occurs in 500 ^0C and higher temperatures (Δw = 69.66%). This indicates that all the organic additives are removed from powders by annealing at 500 ^0C.

The XRD patterns of prepared SnO_2 nano-powders at different annealing temperatures are shown in Fig.1 powder annealed at 200^OC is less crystalline with a large amorphous background. Those annealed at 500^0C and 700 ^0C are completely crystallized. Thus, crystallization starts at 500 ^0C and amorphous background falls down. XRD peaks were indexed using least square method using standard computer program POWDMULT which gives pure tetragonal SnO_2 single phase with a = 4.7383A^0, c = 3.1948A^0. Width of the peaks decreases with increasing annealing temperature. The mean size of nano-particles was also calculated as 23 nm.

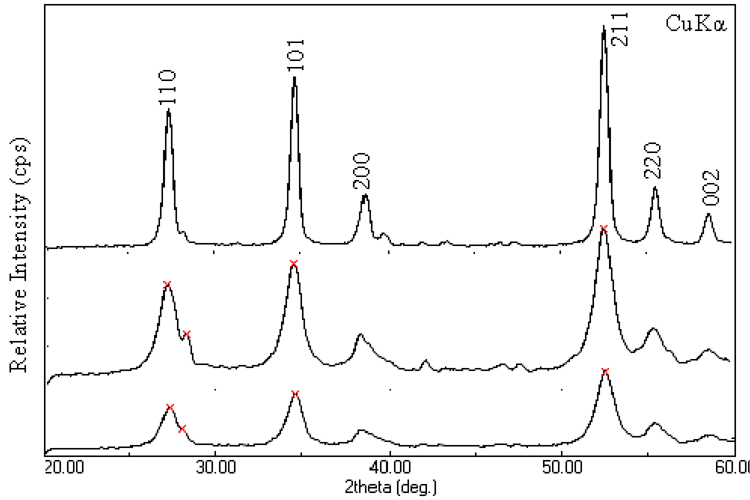

Figure 1 XRD pattern of SnO_2 nano powders annealed at 200, 500 and 700^0C (from bottom to top).

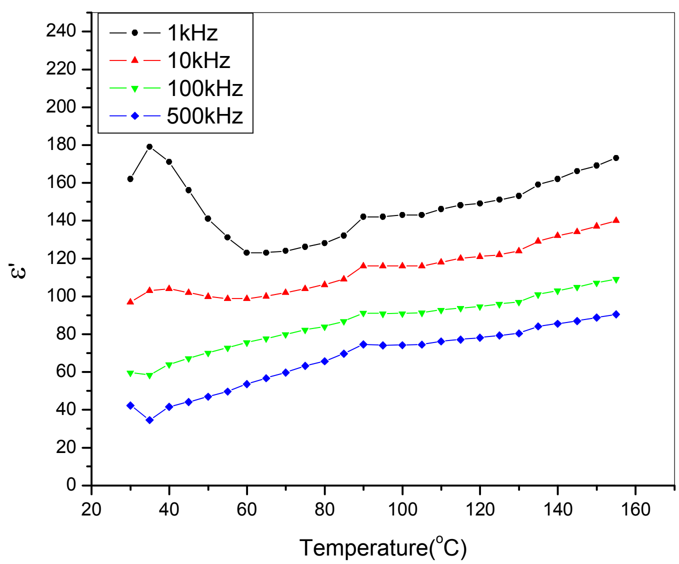

Figure 2 Temperature dependence of real part of dielectric constant (ε') in SnO_2 nanoparticles.

Fig 2 shows the temperature dependence of dielectric constant. Dielectric constant increases monotonically with temperature and decreases with frequency. A small frequency dependent low temperature dielectric anomaly is apparent that may be associated with space charges at interface.

The relatively higher values of dielectric constant than obtained for bulk SnO2 is also observed indicating that the nano material may be a better capacitor sensor element.

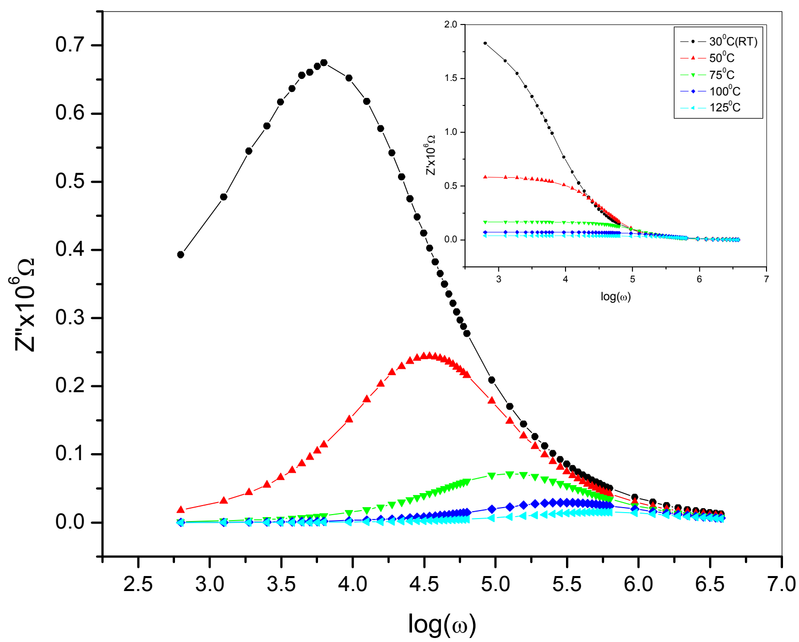

Figure 3 Frequency dependence of imaginary part of Impedance (Z”). Inset shows the same for real part (Z’). Temperatures are same for both graphs as shown in the inset.

The frequency dependence impedance response is shown in figure 3. Z’ decreases with increasing frequency and temperature. The high-frequency impedance is however temperature independent.. As the temperature increases, the peaks start appearing in Z”. The peaks shift towards higher frequency with increasing temperature showing that the resistance of nano particles decreasing. .Also the magnitude of Z” decreases with increasing frequency, indicating the behavior as semiconductor one.

The relation between Z’ and Z” for different temperatures is shown in fig 4. A single semi-circular arc is observed for all temperatures. This confirms the major contribution to impedance coming from grains. Further, the arc crosses the Z’-axis at lower values with increasing temperature, showing that the grain resistance decreases with temperature.

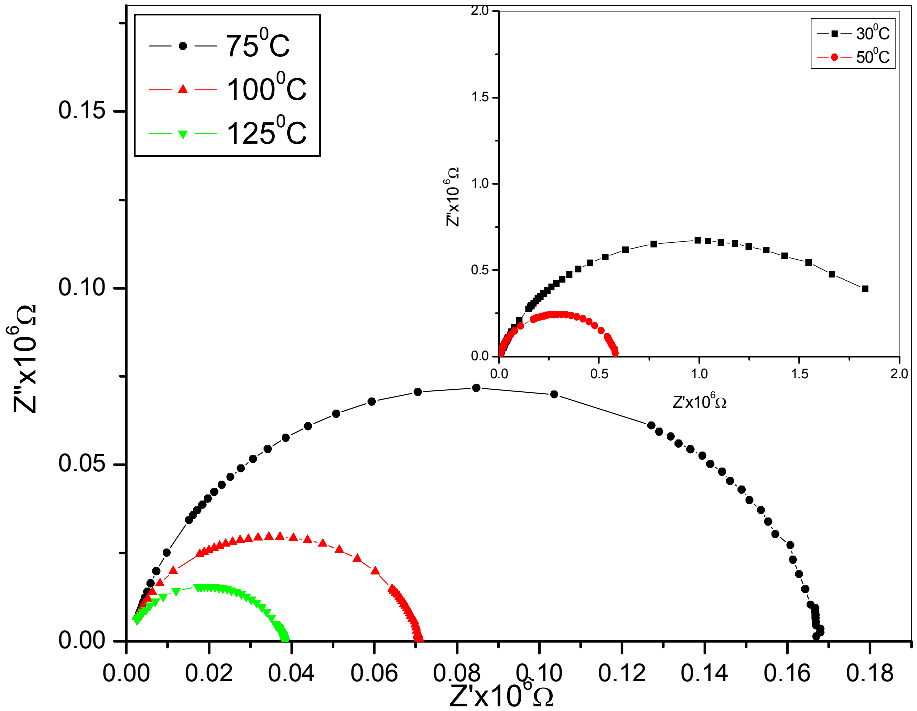

Figure 4: Nyquist plot between Z' and Z" at representative temperatures.
Inset shows the same at lower temperatures.

4. Conclusions

Nano-crystalline SnO_2 particles have been synthesized by chemical route using a simple starting materials consisting of $SnCl_2.2H_2O$ and NH_4OH as complexing and ethanol as polymerization agents. The structural properties of the prepared tin oxide nano-powders annealed at different temperatures (200^0C–700^0C) have been characterized by X-ray diffraction (XRD).The XRD pattern shows tetragonal phase in the nano powder with lattice parameter a=4.7383A0 c= 3.1948 A^0. The Size of the prepared material is estimated to be ≈23 nm crystals. Impedance spectroscopy measurements taken in the temperature range between RT and 150 ^{0}C show a clear dependence of the measured impedance on the particle size. The impedance analysis reveals grain resistance decreasing with temperature as expected for a semi conducting material.

References

1. M. Bhagwat, P. Shah ad V. Ramaswamy, *Mater. Lett.*, **57** (2003) 1604.
2. B. G. Lewis and D. C. Paine, *MRS Bull.*, **25 (8)** (2000) 22.
3. H. L. Hartnagel, A. L. Dawar, A. K. Jain and C. J. Jagadish, Semiconducting Transparent Thin Films, *IOP, Bristol*, 1995.
4. M. Shoyama and N. Hashimoto, *Sensor. Actuat.*, **B 93** (2003) 585.
5. X. Yuan, L. Cao, H. Wan, G. Zeng and S. Shiquan, *Thin Solid Films*, **33** (1998) 327–329.
6. L. R. B. Santos, T. Chartier and C. Pagnoux, *J. Eur. Ceram. Soc.*, **24** (2004) 3713.
7. C. Premakumara, M. Kakihana, M. Yoshimura, *Solid State Ionics*, **108** (1998) 23.