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Role of temperature on the properties of SnO_2 Nanoparticles synthesised by Sol-Gel Process

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Abstract: SnO_2 nanoparticles were synthesized using Sol-Gel process at different annealing temperatures ranging from 200°C to 400°C by maintaining the pH value of the gel at 8. The role of temperature on the structural, and optical properties of SnO_2 nanoparticles were studied using X-ray diffraction (XRD), Fourier Transform Infra-red Spectroscopy (FTIR) & UV-Vis Spectroscopy. X-ray diffraction study reveals the formation of Nano-metre scale SnO_2 particles with tetragonal rutile structure in the range of 1.5nm to 5nm. FTIR study indicates the structural confirmation of SnO_2 nanoparticles. UV study shows that with increase in temperature, the optical band gap decreases with splitting of energy. The results obtained at different temperatures are compared with the freshly prepared sample. It was concluded that the size and hence the band gap of the Tin oxide nanoparticle can be tuned in a controllable manner and can be used for various potential applications.
Keywords: Tin oxide nanoparticles; Sol-Gel method; annealing and pH value.

Introduction and Experimental:

Nanoparticles effectively form a bridge between bulk materials and atomic or molecular structures. The particle size of the material has significant influence on its physical and chemical properties. Among various classes of Nanoparticles (Metals, Semiconductors and Insulators), semiconductor particles have attracted more interests because of their size-dependent optical & electrical properties. Tin oxide is an important n-type semiconductor with a wide band gap of 3.6-3.8 eV. It has many potential applications such as optoelectronic devices, fabricating solar cells, electrochemical applications, electrode materials for lithium batteries, catalysts for redox reactions and as gas sensing material for gas mixture such as CO, H_2 and NO. Many methods have been developed for the synthesis of SnO_2 nanoparticles [1-2]. Among Various methods, Sol-gel process is an effective way of preparing chemically homogeneous, high-purity and phase-pure powders at lower temperature. In the present paper, the role of temperature on the structural, optical & thermal properties of SnO_2 nanoparticles synthesized by Sol-gel process at different annealing temperatures ranging from 200°C to 400°C by maintaining the pH value at 8 were investigated and the results obtained were compared with that of the freshly prepared sample.

Hydrous Tin (IV) chloride ($SnCl_4.5H_2O$. (A.R)) was dissolved in deionised water. Certain amount of Ammonia solution was added into the mixture under constant stirring. The resulting solution was aged at room temperature for 24 hrs. The pH value was set at 8 by adding ammonia solution [3] .The obtained opal gel was washed with deionised water and ethanol several times to remove impurities. Then it was dried at 80^0C for several hours. The resulting product was grind well using mortar and pestle. Then it was annealed at temperatures 200°C, 300°C & 400°C for 2 hrs.

Results and Discussion:

XRD Analysis

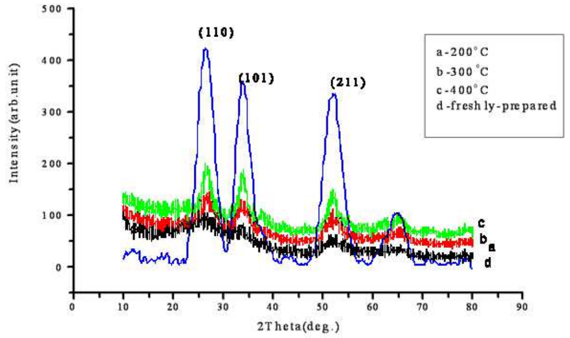

Fig. 1. XRD pattern of SnO_2 nanoparticles prepared at pH=8 for various temperatures

The XRD pattern of SnO_2 nanoparticles prepared at pH=8 for various temperatures is shown in Fig.1. The observed pattern has prominent peaks at (110),(101) & (211) phase. This is in excellent agreement with a reference pattern (Powder diffraction file No.21-1250.) The crystallite size of the as-prepared powder is calculated using Debye Scherrer formula,

D = 0.9 λ / β Cosθ.

The average crystallite size is found to be 1.96nm, 3.97nm & 4.87nm for samples annealed at 200°C, 300°C & 400°C respectively. The average crystallite size of the freshly prepared sample is found to be 1.48nm. The results obtained revealed that increase in annealing temperature increases the particle size. To the best of our knowledge, the crystallite size obtained by maintaining the pH value 8 is found to be smaller than those obtained by other methods of synthesis [2,3] and also those obtained [4] at pH value 9.5.

FTIR Analysis

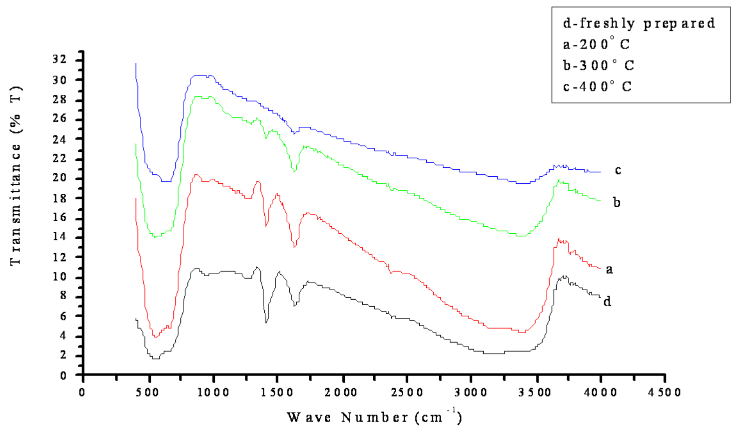

Fig. 2 FTIR spectra of SnO_2 nanoparticles prepared at pH=8 for various temperatures

The peaks at about 550cm^{-1} were attributed to Sn-OH group and peaks at about 660 cm^{-1} were attributed to the stretching modes of Sn-O-Sn. As the sample is annealed to 400°C, the Sn-OH band disappears and Sn-O-Sn band became stronger [5].

$$SnOH + SnOH \rightarrow Sn\text{-}O\text{-}Sn + H_2O$$

In the freshly prepared sample, the peaks at about 1401 cm^{-1} & 3135 cm^{-1} are attributed to absorption of water and ammonia. As the annealing temperature is increased, the intensity of the water bands decreases and at about 400°C, the bands attributed to water & NH_3 disappears. The intensity of the bands at 3394 cm^{-1} -3409 cm^{-1} & 1635 cm^{-1} -1619 cm^{-1} is greatly reduced as the temperature increases. The broad band around 3394 cm^{-1} -3409 cm^{-1} region is due to the stretching vibration of -H bond. This band is due to the OH groups and absorbed water bound at the SnO_2 surface. The peak at 1635 cm^{-1} -1619 cm^{-1} is attributed to the bending vibration of water molecules, trapped in the SnO_2 sample.

UV-Visible Analysis

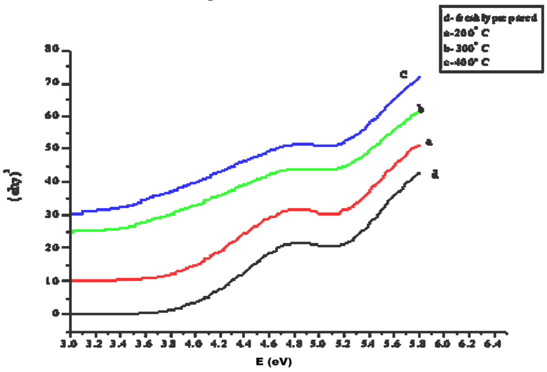

Fig. 3 Energy gap spectra of SnO_2 nanopartiles

The optical band gap energy (E_g) of the semiconductor is calculated from Tauc relation. A plot of $(\alpha h\nu)^2$ versus hν shows intermediate linear region. The extrapolation of the linear part can be used to calculate the E_g from the point of intersection with hν axis as shown in Fig. 3. The resultant values of E_g for freshly prepared SnO_2 nanoparticle is found to be about 3.7eV and 4.52 eV with splitting of energy [2, 6]. These two values may be related to the formation of bulk SnO_2 and nanostructures of SnO_2. When the size of SnO_2 nanocrystallites is smaller or comparable to the exciton Bohr radius, the quantum confinement would occur and blue shift in energy is observed. As the annealing temperature increases, the band gap energy decreases. The band gap values are found to be 4.39eV, 4.30eV & 4.24eV for the samples annealed at temperatures 200°C, 300°C & 400°C respectively. The band gap energy is dependent on the particle size as well as on the temperature. This is confirmed from the XRD & UV results. Thus the band gap of the SnO_2 nanoparticle can be tuned in a controllable manner which makes it a suitable candidate for various potential applications.

Acknowledgement

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