JETIR.ORG ISSN: 2349-5162 | ESTD Year : 2014 | Monthly Issue
JOURNAL OF EMERGING TECHNOLOGIES AND INNOVATIVE RESEARCH (JETIR)
An International Scholarly Open Access, Peer-reviewed, Refereed Journal

Synthesis and Characterization of SnO_2 Nanoparticles prepared by Sol Gel Technique.

V. R. Chinchamalatpure.

Assistant Professor, Department of Physics, Hutatma Rashtriya Arts and Science College Ashti, Distt: Wardha, India.442202

Abstract:

Tin oxide nanoparticles have been prepared by conventional sol-gel technique. Tin chloride ($SnCl_2.2H_2O$) was used as tin and zinc sources. Ethylene glycol and alcohol were used as solvents under different synthesis conditions. Transparent SnO_2 sol was prepared after continuous stirring and refluxing at 50^oC. Thin films prepared from SnO_2 sol have also been deposited on glass substrate by spin coating at 500 rpm for 15 seconds and then at 3000 rpm for 20 seconds. The morphological, compositional and structural properties of SnO_2 nanoparticles were characterized by XRD, Scanning electron microscopy (SEM), FT-IR and UV-vis-IR absorption spectrum.

X-ray diffraction (XRD) patterns show that the preferred orientation was (111) with orthorhombic crystal structure. Crystallite size was calculated to be ~30 nm from XRD results. Scanning electron microscopy (SEM) results show the formation of Nano diamonds (~ 40 nm) and Nano rods (~30 nm) after annealing at 300^oC for 60 minutes. It is important to mention here that the preparation of SnO_2 nanostructures without the use of any surfactant and ligand during sol synthesis. Fourier Transform Infrared (FTIR) spectra give peaks due to resonance of vibration, stretching of atoms and bond between them.

Index Terms: Nanoparticles; SnO_2, SEM, XRD, FT-IR.

I. INTRODUCTION:

Nano materials have attracted great interest due to their intriguing properties, which are different from those of their corresponding bulk state. Enormous efforts are being taken towards the development of nanometer sized materials in studies related on one hand to their fundamental mechanism such as the size effect and the quantum effect, on the other hand towards application of these materials.

Nanometer sized material and semiconductor particles have a large potential for industrial applications. Metal oxide semiconductors are low cost and effective gas sensing material. Among the various metal oxide semiconductors, Tin Oxide SnO_2 have been attracting much attention since they are highly conducting, transparent and sensitive to gases. SnO_2 as a n-type semiconductor because of wide-

energy-gap has attracted many considerations, therefore this product was used in many fields such as transparent conducting films, catalytic materials, environmental monitoring, biochemical sensor, lithium rechargeable batteries, dye-sensitized solar cells and ultrasensitive gas sensors [1-4].

Many processes have been developed to the synthesis of SnO_2 nanostructures, e.g., spray pyrolysis [5], hydrothermal methods [6-8], evaporating tin grains in air [9], chemical vapor deposition [10], thermal evaporation of oxide powders [11], rapid oxidation of elemental tin [12], the sol–gel method [13] etc. Davar et al. [14] reported the synthesis of SnO_2 nanoparticles by thermal decomposition using [bis(2-hydroxyacetophenato) tin(II)], [$Sn(HAP)_2$], as precursor. Salavati-Niasari et al.[15] synthesized zinc blend ZnS nanoparticles by a thioglycolic acid ($HSCH_2COOH$)-assisted hydrothermal technique via the reaction between a new inorganic precursor [bis(2-hydroxyacetophenato) zinc(II)], [$Zn(HAP)_2$] and thioacetamide (CH_3CSNH_2). Gnanam and Rajendran [16] synthesized Nano crystalline tin oxide powders of about 15 to 20 nm in size using different surfactants. such sodium dodecyl sulphate and polyethylene glycol via hydrothermal reaction at 150°C for 12 hours and studied their structural and photoluminescence properties. In addition to indepth exploration of gas sensing mechanisms [17]. Metal oxide semiconductor materials such as SnO_2, ZnO, TiO_2, WO_3, Fe_2O_3, ZrO_2, Cr_2O_3, $BaTiO_3$, Ga_2O_3 etc. have been well reported as gas sensors in the form of thick film [18]. The SnO_2 is used because of their long term stability, small size, light weight, low cost, good mechanical strength and high reliability. It has a strong physical and chemical interaction with adsorbed species and thermal stability in air up to 500 ^{0}C [19]. The changes in the properties of SnO_2 due to gas adsorption are related to the nonstoichiometry, average co-ordination number per grain and the neck size effect in the functional material. As such there is no specific report relating to its structure and sensing properties. Catalysts like Pt, Pd, Ag, Ru and CuO often added to the base material to improve the gas sensitivity and selectivity [20,21].

In the present work, I have studied the structural and properties of SnO_2 nanoparticles. This material was characterized by using XRD, SEM, FTIR and UV-vis-IR absorption spectrum of SnO_2 nanoparticles with different hydrolysis rate.

II. EXPERIMENTAL DETAILS:

SnO_2 nanoparticles were synthesized by using Sol-Gel method. All the chemicals used for the preparation were of analytical grade. It includes tin chloride hydrate $SnCl_2.2H_2O$ was used as starting material. A solution of 0.1 mole % $SnCl_2$ in ethylene glycol was prepared by dissolving appropriate amounts of $SnCl_2$ under vigorous stirring at 60°C until colorless and transparent sol was obtained.

The powder of SnO_2 has been prepared and taken in 100 ml beaker and adds 3- 4 ml. 1% poly vinyl alcohol. The mixture is sticky, dry the mixture with a natural process. This mixture is added with 150 ml. millipore water.

The solution was allowed to centrifuge in presence of water and acetone to remove impurities, for the solution and allowed to dry at room temperature. Dried powder of SnO_2 was kept in stainless steel autoclave for 24 hours at 150^{0}C and calcinaised at 400^{0}C for 2 hours in a muffle furnace. The dried powder

of SnO_2 is used for the characterization by XRD, SEM, FTIR. etc. Before gelation process thin films were also prepared on glass substrate by spin coating. Samples were dried at room temperature for 24 hours and then annealed at 300°C for 1 hour.

II. RESULTS AND DISCUSSION:

3.1 Structural Characterization:

Crystallographic structure along with the phase variation and crystallite size of SnO_2 samples was studied by Rigaku D-MAX/IIA X-ray Diffractometer (XRD). CuKα (Ni filtered) radiations (λ =1.5405 A.U.) were used to obtain the XRD pattern. Surface morphology was observed using Hitachi S-3400N Scanning Electron Microscope (SEM).

The X-ray diffraction study was undertaken. X-Ray diffraction analysis of SnO_2 samples were carried out in the range 20-80^0 range using CuKα radiation. Figure 1. shows an XRD pattern of SnO_2 sample plotted in the range 20-80^0 (2θ) verses intensity having several peaks of SnO_2 indicating random orientation for the tetragonal rutile nature and measured interplaner distance agreed with the value reported for SnO_2 in literature, which gives a highest peak at 26.78 degree on calculating the crystal size by Debye Scherer formula, it is found that the size is about 33.4 nm.

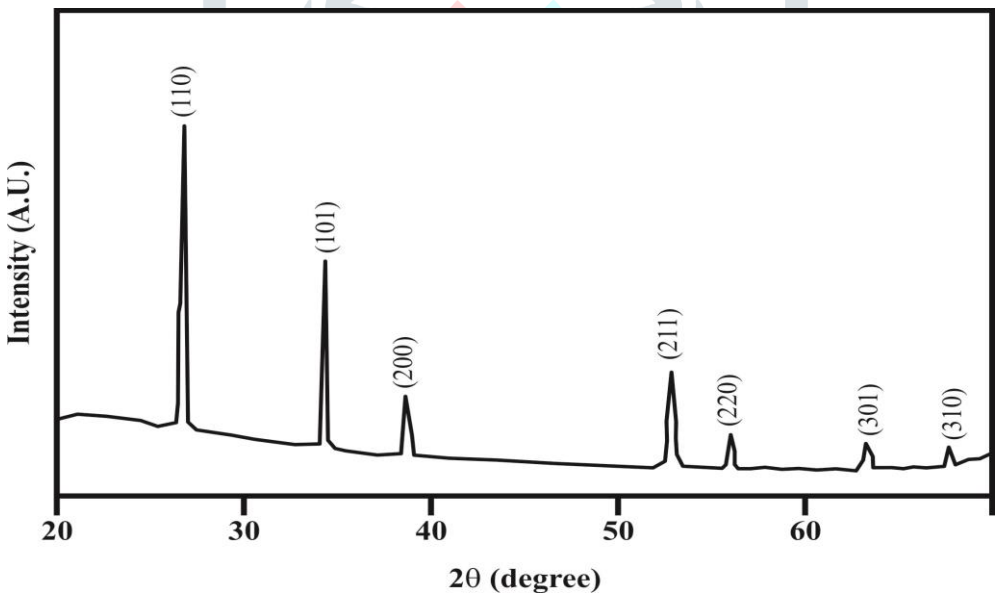

Fig.1. shows an XRD pattern of SnO_2 sample.

The observed peaks match well with the reported JCPDS data of SnO_2, confirming the tetragonal rutile nature. The higher peak intensities of an XRD pattern is due to the better crystallinity and bigger grain size. The average grain size calculated by using formula $D = \lambda n / \beta Cos\theta$

Where D is the crystalline size and λ is the wavelength β is FWHM and θ is the measured angle.

3.2 Surface Morphology:

The surface morphology of the powder was analyzed using a Scanning electron microscope. The model JEOL, JSM-6360 was used for the determination of morphology of nanoparticle. Fig. 2 depicts the Scanning electron micrograph (SEM) of the prepared SnO_2 nano powder annealed at 400^{0}C for 2 hours. The image indicates that the particles are spherical in nature. Larger particles in this figure may be aggregates of the smaller particles. The surface of SnO_2 nanostructure exhibits a highly corrugated surface, where voids

between the crystalline increase the specific surface area, which is a key feature for high sensitivity of gas sensors.

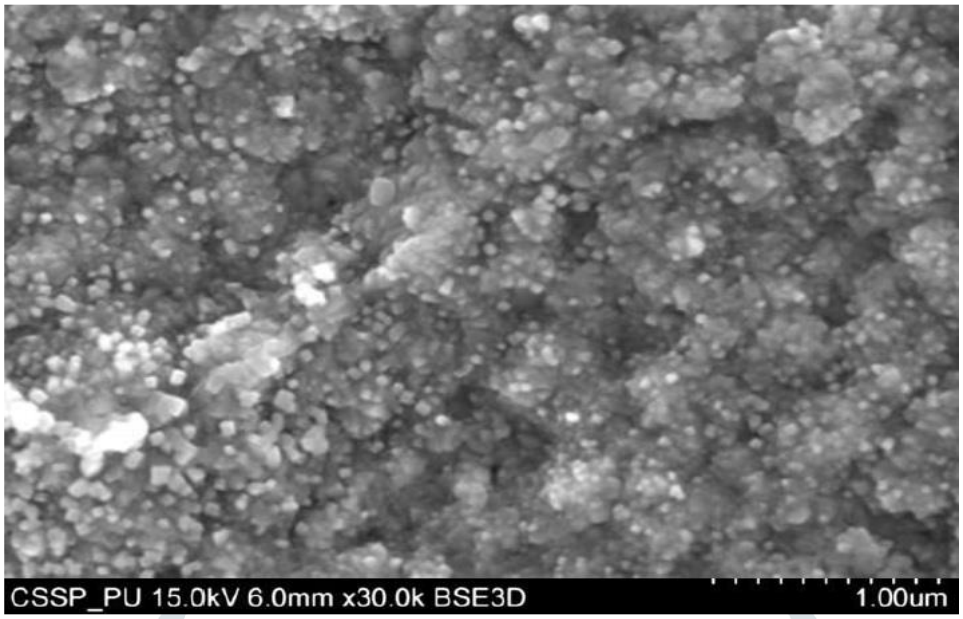

Fig.2 shows Scanning Electron Microscopy (SEM) of SnO_2 nanostructures at 300°C.

3.3 FTIR Spectra :

The FTIR spectra are analyzed between absorption and wavelength, these vibrational spectra gives peaks due to resonance of vibration, stretching of atoms and bond between them in Fig. 3. Molecular bonds vibrate at various frequencies depending on the elements and the type of bonds. For any given bond, there are several specific frequencies at which it can vibrate. According to quantum mechanics, these frequencies correspond to the ground state at lowest frequency and several excited states at higher frequencies. One way to cause the frequency of a molecular vibration to increase is to excite the bond by having it absorb light energy. For any given transition between two states the light energy must exactly equal the difference in the energy between the two states usually ground state (E_0) and the first excited state (E_1).

Difference in the energy state = Energy absorbed

$E_1 - E_0 = h c / \lambda$

Where, h is the Plank's constant, c is the velocity of the light, λ is the wavelength of the light.

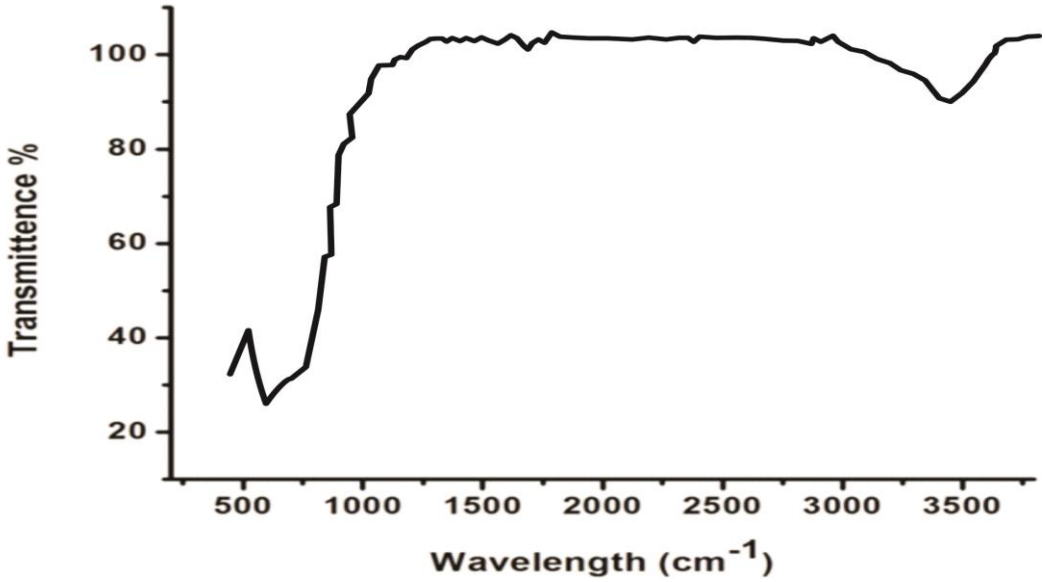

Fig. 3 shows the FTIR pattern of SnO_2

For crystalline SnO_2, optical transition has been shown to be direct. The variation in the absorption coefficient as a function of photon energy for allowed direct is given by

$$\alpha(h\nu) = A(h\nu - Eg)^{1/2}$$

where, α is the absorption coefficient, A is a constant, h is Planck's constant, ν is the frequency, and Eg is the band gap energy.

3.4 UV-vis-IR absorption spectrum:

The absorption spectra in the UV-vis-IR range of SnO_2 nanoparticles with different hydrolysis rate are presented in Fig.4.

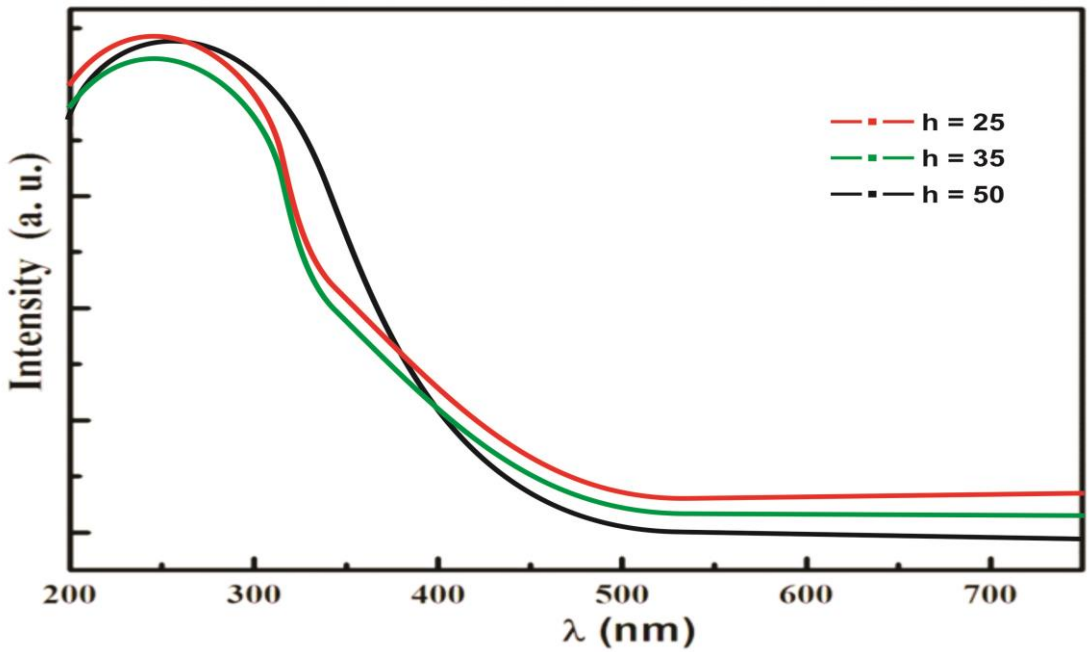

Fig. 4. UV-vis-IR absorption spectrum of SnO_2 nanoparticles with different hydrolysis rate.

All absorption curves exhibit an intense absorption in the 200 - 400 nm wavelength range and an absorption edge between 300 and 350 nm, owing to the relatively large exciton binding energy.

IV. CONCLUSIONS:

The present study illustrates that Sol-Gel Spin Coating method which produce single phase material at lower temperature and shorten the synthesis time. X-Ray diffraction (XRD) result shows that the obtained SnO_2 nanoparticles were composed of tetragonal lattice nature with high crystallinity. Scanning electron microscopy (SEM) result showed that grains are uniformly distributed and the particles are spherical in nature. The average particle size was obtained 35 nm. The FTIR spectra gives peaks due to resonance of vibration, stretching of atoms and bond between them. In UV–vis–IR absorption spectrum of SnO_2 nanoparticles all absorption curves exhibit an intense absorption, owing to the relatively large exciton binding energy.

V. REFERENCES:

[1] H. Liu, Sh. Gong, Y. Hu, J. Zhao, J. Liu, Zh. Zheng, D. Zhou, 2009. Synthesis and Characterization of SnO_2 nanoparticles by a facile Precipitation Method, Ceram. International. 35: 961-966.

[2] X. Chu, Y. Han, S. Zhou, H. Shui, 2010. Trimethylamine Sensing Properties of Nano-SiO_2 prepared using Microwave Heating Method. Ceram. International. 36: 2175-2180.

[3] Y. Tan, Ch. Li, Y. Wang, J. Tang, X. Ouyang, 2008. Fast-response and high sensitivity gas sensors based on SnO_2 hollow spheres. Thin Solid Films, 516: 7840-7843.

[4] Y. Wang, Ch. Ma, X. Sun, H. Li,2002. Synthesis and Characterization of SnO_2 nanoparticles by Co-Precipitation Method , Inorg. Chem. Commun. 5: 751-755.

[5] Paraguay-Delgado, F.,Antúnez-Flores, W., Miki-Yoshida, M., Aguilar-Elguezabal, A, Santiago, (2005).Structural analysis and growing mechanisms for long SnO_2.Thin Solid Films, 16: 688-696.

[6] Cheng, B., Russell, J.M., Shi, W., Zhang, L., Samulski, E.T. , 2004. Large-scale, Solution phase growth of single-crystalline SnO_2 nanorods. Journal Am. Chem. Soc. 126: 5972-5982.

[7] Du, F., Guo, Z., Li, G. 2005.Hydrothermal synthesis of SnO_2, hollow microspheres. Mater. Lett. 59, 2563-2572.

[8] Fujihara, S., Maeda, T., Ohgi, H., Hosono, E., Imai, H., Kim, S. 2004. Hydrothermal routes to prepare Nano crystalline mesoporous SnO_2 having high thermal stability. Langmuir 20: 6476- 6488.

[9] Duan, J., Yang, S., Liu, H., Gong, J., Huang, H., Zhao, X., Zhang, R., Du, Y. 2005.Single crystal SnO_2 , zigzag nanobelts. Journal of American Chem. Soc. 127: 6180-6191.

[10] Liu, Y., Koep, E., Liu, M. 2005. A highly sensitive and fast-responding SnO_2 sensor fabricated by combustion chemical vapor deposition. Chemical Mater 17: 3997-4010.

[11] Dai, Z.R., Gole, J.L., Stout, J.D., Wang, Z.L. 2002.Tin oxide nanowires, nanoribbons, and nanotubes. J. Phys. Chem. B 106:1274.

[12] Hu, J.Q., Ma, X.L., Shang, N.G., Xie, Z.Y., Wong, N.B., Lee, C.S., Lee, S.T. 2002.Large scale rapid oxidation synthesis of SnO_2 Nano ribbons. J. Phys. Chem. B 106: 3823.

[13] Pourfayaz, F., Khodadadi, A., Mortazavi, Y., Mohajerzadeh, S.S. 2005.SnO_2 sensor selective to ethanol in presence of CO, LPG and CH_4. Sensors Actuators B108: 172-181.

[14] Davar, F., Salavati-Niasaria, M., Fereshteh, Z.2010.Synthesis and characterization of SnO_2 nanoparticles by thermal decomposition of new inorganic precursor. J. Alloys Compd. 496(1-2): 638-643.

[15] Salavati-Niasari, M., Davar, F., Seyghalkar, H., Esmaeili, E., Mir, N.2011.Synthesis and characterization of SnO_2,nanoparticles by thermal decomposition of new inorganic precursor. Crystal. Eng. Comm. 13: 2948-2960

[16] Gnanam, S., Rajendran, V. 2012. Anionic, cationic and nonionic surfactants-assisted hydrothermal synthesis of tin oxide nanoparticles. Digest Journal of Nanomaterials and Biostructures 5(2), 6: 547-558.

[17] A. Gurlo, R. Riedel, 2007. In situ and operando spectroscopy for assessing Mechanisms of gas sensing, Angew. Chem. Int. Ed., 46: 3826-3848.

[18] V. Guidi, M. A. Butt Uri, M. C. Carrotta and B. Cavicchi, 2002.Gas sensing Through Thick Film Technology, Sensors and Actuators *B*, 84: 72-77.

[19] S. H. Park, Y. C. Son, W. S. Suib and K E Creasy, 2008.Tin oxide Films made by physical vapor deposition thermal oxidation, Chem. Mater., 10, 2389-2398.

[20] S. Matsushima, T. Maekawa, J. Tamaki, N. Miura, N. Yamazoe, 2009.Roll of additives on alcohol sensing by semiconductor gas sensor, Chemical Letters, 11:845-848

[21] C. Xu, J. Tamaki, N. Miura and Y. Yamazoe, 2009. Relation between gas sensitivity and microstructure of Porous SnO_2, Journal of Electrochemical Soc. Japan, 58:1143-1148.