

Mixture and Description of SnO₂ Nanoparticles complete by Sol Gel System.

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Abstract:

Thin films prepared from SnO₂ 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.

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°C for 60 minutes.

Index Terms: Nanoparticles; SnO₂, SEM, XRD, FT-IR.

INTRODUCTION

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.

Metal oxide semiconductors are low cost and effective gas sensing material. Among the various metal oxide semiconductors, Tin Oxide SnO₂ have been attracting much attention since they are highly conducting, transparent and sensitive to gases. SnO₂ 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 SnO₂ nanostructures, e.g., spray pyrolysis [5], hydrothermal methods [6-8], evaporating tin grains in air [9], chemical vapor deposition [10], thermal evaporation Davar et al. [14] reported the synthesis of SnO₂ 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 (HSCH₂COOH)-assisted hydrothermal technique via the reaction between a new inorganic precursor [bis(2-hydroxyacetophenato) zinc(II)], [Zn(HAP)₂] and thioacetamide (CH₃CSNH₂). 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. Metal oxide semiconductor materials such as SnO₂, ZnO, TiO₂, WO₃, Fe₂O₃, ZrO₂, Cr₂O₃, BaTiO₃, Ga₂O₃ etc. have been well reported as gas sensors in the form of thick film [18]. The SnO₂ 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 °C [19]. The changes in the properties of SnO₂ 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].

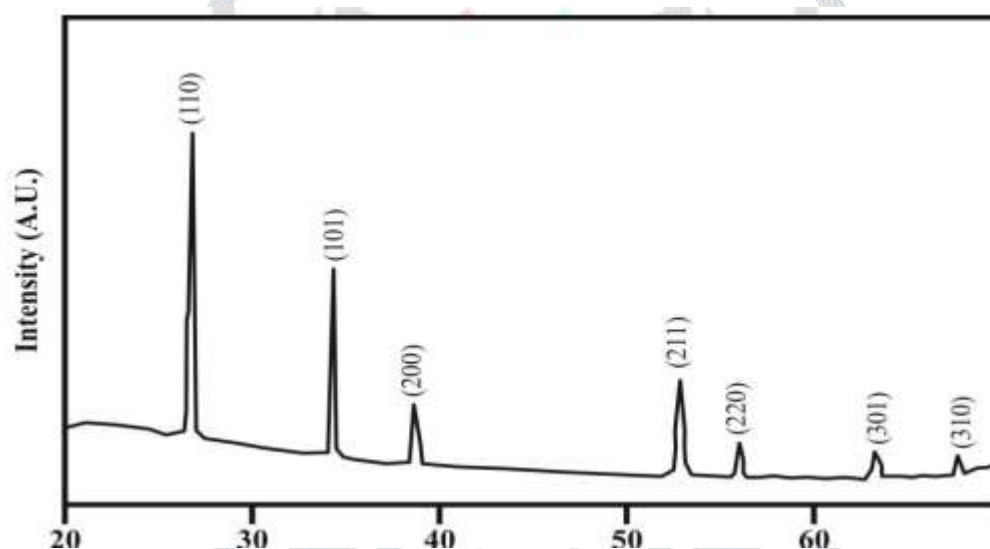
EXPERIMENTAL DETAILS:

SnO₂ nanoparticles were synthesized by using Sol-Gel method. All the chemicals used for the preparation were of analytical grade. It includes tin chloride hydrate SnCl₂.2H₂O was used as starting material. A solution

of 0.1 mole % SnCl₂ in ethylene glycol was prepared by dissolving appropriate amounts of SnCl₂ under vigorous stirring at 60°C until colorless and transparent sol was obtained. 1% poly vinyl alcohol. The mixture is sticky, dry the mixture with a natural process. 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 SnO₂ was kept in stainless steel autoclave for 24 hours at 1500C and calcinaised at 4000C for 2 hours in a muffle furnace. The dried powder of SnO₂ is used for the characterization by XRD, SEM, FTIR. etc. Samples were dried at room temperature for 24 hours and then annealed at 300°C for 1 hour.

RESULTS AND DISCUSSION

Crystallographic structure along with the phase variation and crystallite size of SnO₂ samples was studied by Rigaku D-MAX/IIA X-ray Diffractometer (XRD). CuK α (Ni filtered) radiations ($\lambda = 1.5405$ A.U.) were used to obtain the XRD pattern. The X-ray diffraction study was undertaken. X-Ray diffraction analysis of SnO₂ samples were carried out in the range 20-800 range using CuK α radiation. Figure. shows an XRD pattern of SnO₂ sample plotted in the range 20-800 (2θ) verses intensity having several peaks of SnO₂ indicating random orientation for the tetragonal rutile nature and measured interplaner distance agreed with the value reported for SnO₂ 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.

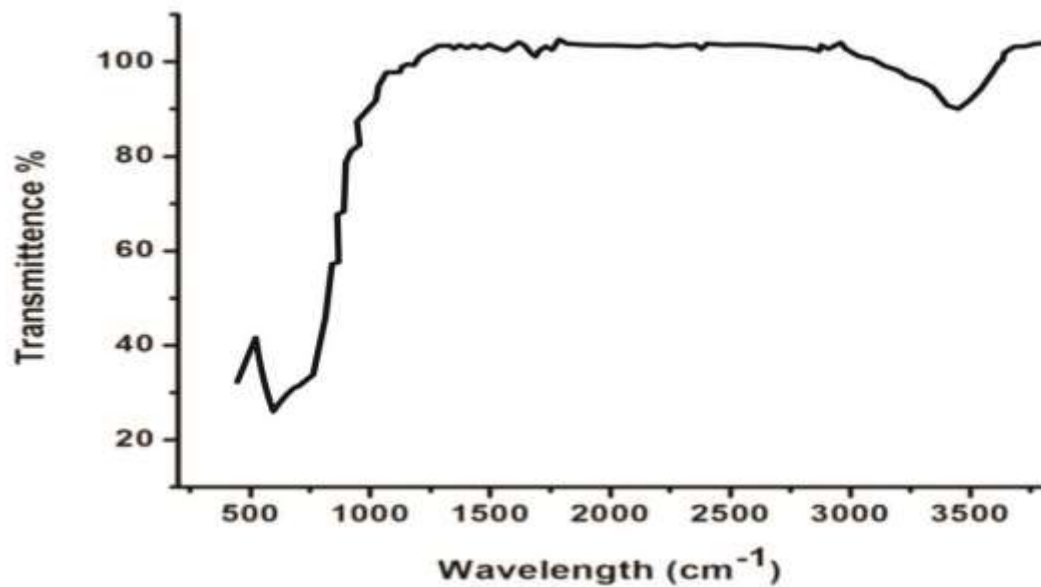


Surface Morphology:

The model JEOL, JSM-6360 was used for the determination of morphology of nanoparticle. Fig. depicts the Scanning electron micrograph (SEM) of the prepared SnO₂ nano powder annealed at 4000C for 2 hours. Larger particles in this figure may be aggregates of the smaller particles. The surface of SnO₂ 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.

FTIR Spectra :

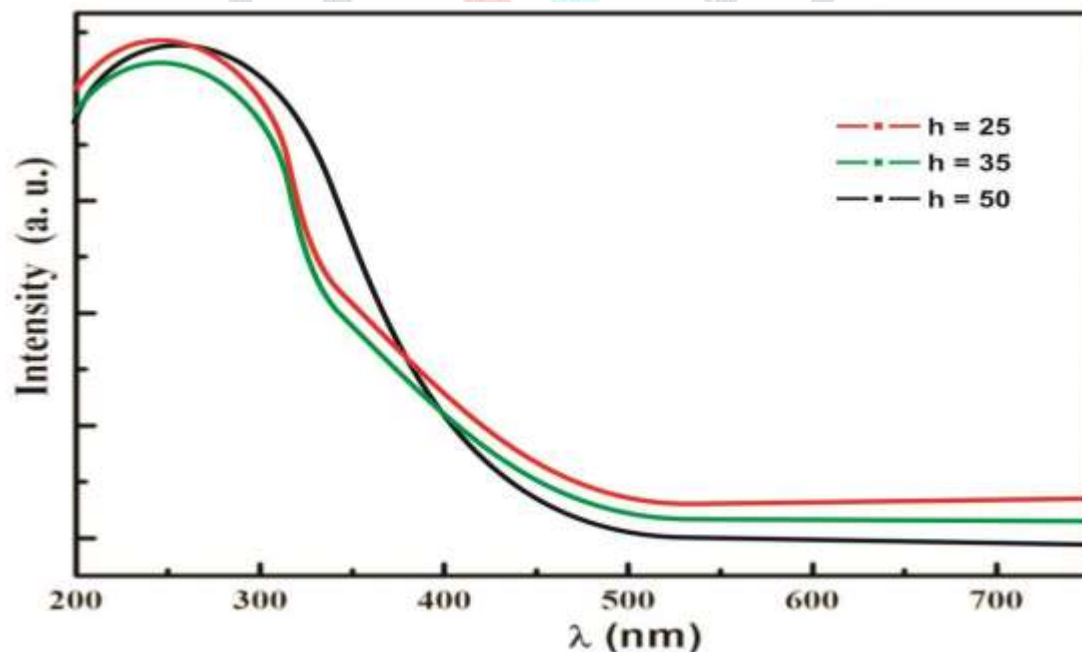
The FTIR spectra are analyzed between absorption and wavelength, these vibrational spectra gives peaks due to resonance of vibration, stretching of atoms and bond between them in According to quantum mechanics, these frequencies correspond to the ground state at lowest frequency and several excited states at higher frequencies. 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 (E₀) and the first excited state (E₁). Difference in the energy state = Energy absorbed E₁ - E₀ = $h c / \lambda$ Where, h is the Plank's constant, c is the velocity of the light, λ is the wavelength of the light.



shows the FTIR pattern of SnO₂

For crystalline SnO₂, 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 $\alpha(h\nu) = A(h\nu - E_g)^{1/2}$ where, α is the absorption coefficient, A is a constant, h is Planck's constant, ν is the frequency, and E_g is the band gap energy.

UV-vis-IR absorption spectrum:



UV-vis-IR absorption spectrum of SnO₂ nanoparticles with different hydrolysis rate.

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 SnO₂ 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 FTIR spectra gives peaks due to resonance of vibration, stretching of atoms and bond between them. In UV-vis-IR absorption spectrum of SnO₂ nanoparticles all absorption curves exhibit an intense absorption, owing to the relatively large exciton binding energy.

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