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Synthesis of SnO₂ Nanoparticles by Chemical Precipitation Method and their Characterization

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ABSTRACT: The chemical precipitation method was successfully used to synthesized SnO₂ nanoparticles powder. X-ray diffraction (XRD) technique was used to examine the structural characteristics of (SnO₂) nanocrystalline powder. UV-Visible absorption spectroscopy was used to investigate the optical properties. The X-ray analysis shows that the obtained powder is SnO₂ with tetragonal crystalline structure and the crystalline size in the range of 8-10nm. The SPM investigation reveals that the average particles size is 73nm. The optical band gap values of SnO₂ nanoparticles were calculated to be about 4.3eV in the temperature 550 ° C, comparing with that of the bulk SnO₂ 3.78eV, by optical absorption measurement.

KEYWORDS: SnO₂nanoparticles, Band gap, X-ray diffraction, Morphology, Optical Properties.

I. INTRODUCTION

Nano materials have recently research attention due to their unique electrical, physical, chemical, and magnetic properties. These materials behave differently from bulk semiconductors. With decreasing particle size the band structure of the semiconductor changes; the band gap increases and the edges of the bands splits into discrete energy levels. These so-called quantum size effects occur [1-5]. These quantum size effects have stimulated great interest in both basic and applied research. Due to its many valuable properties, including its wide band gap ($E_g = 3.64$ eV, 330 K), n-type conductivity, and high transparency in the visible range (> 80%), tin oxide (SnO₂) is one of the significant metal oxides. It can be applicable for a variety of applications such as solid-state gas sensors, flat panel displays, and solar energy cells [1, 2].

Tin oxide (SnO₂) is one of the most interesting materials to be investigated today. This is for the reason that SnO₂ is a well-known n-type semiconductor with a wide band gap of 3.6-3.8 eV [6-8] and for its potential applications in transparent conductive electrode for solar cells a gas sensing material for gas sensors devices, photoconductive devices and transparent conducting electrodes in liquid crystal display, gas discharge display, lithium-ion batteries, etc[9- 14]. So many processes have been developed to the synthesis of SnO₂ nanostructures, e.g., spray pyrolysis, hydrothermal methods, thermal evaporation of oxide powders, sol-gel method and chemical vapor deposition [15-20].

In the present work the structural, morphological, and optical characteristics of SnO₂ nanopowder were studied. With the help of several characterization methods like X-ray diffraction (XRD), scanning electron microscopy (SEM), and UV-VIS spectroscopy, the characteristics of these SnO₂ nanocrystallites have been investigated.

II. EXPERIMENTAL

2.1 Materials

Without additional purification, SnO₂ nanoparticles were created using Analytical Reagent (A.R.) grade chemicals including (SnCl₂.2H₂O) and ammonia (Make: Loba Chemie Pvt. Ltd).

2.2 Method

SnO₂ nanopowders were prepared by dissolving of 2 g (0.1 M) stannous chloride dehydrate (SnCl₂.2H₂O) in 100 ml distilled water. After complete dissolution, ammonia solution was added to the above solution by drop wise under stirring. The resulting solution were filtered and dried at 80°C for 24 hours in order to remove water molecules. Finally, tin oxide nanopowders were formed at 550°C for 2h.

2.3 Characterization

The samples were characterized by X-ray diffraction(XRD) using (XRD -6000). The surface morphology of the samples was observed by Scanning probe Microscope (SPM) by using CSPM AA3000. Optical absorption spectra of the samples were taken with OPTIMA SP-3000 UV-VIS Spectrometer. The room temperature photoluminescence (PL) spectra of SnO₂ were recorded with SL 174 Spectrofluorometer.

III. RESULTS AND DISCUSSION

A. Structural characteristics of (SnO₂) powder

The structural properties of SnO₂ nano-powder were studied by X-ray diffraction measurements (Bruker D-8 advance diffractometer, Billerica, MA) using the Cu K α ($\lambda = 1.5406 \text{ \AA}$) as a radiation source, operated at 40kV and 30 mA with a scan rate of 0.02°/s over the range of 10°-80°. The average crystallite size $d(hkl)$ of all crystal planes for SnO₂ powder was estimated from the classical Scherrer formula [14]:

$$D = \frac{K\lambda}{\beta \cos \theta} \quad (1)$$

where K is the shape factor usually has a value 0.9, λ is the X-ray wavelength and θ the Bragg angle and β gives the full width of the half maxima (FWHM)

The powder X-ray diffraction analysis of the SnO₂ sample was performed in order to identify the phase, crystal structure and the average grain size was found to be the range of 8-10nm. The X-ray diffraction pattern of SnO₂ powder sample by chemical precipitation method is as presented in Figure 2.

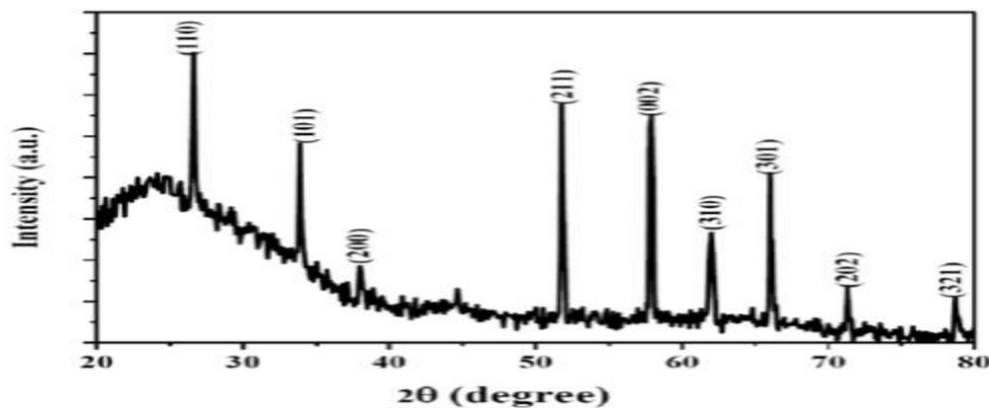


Fig. 1. XRD pattern of SnO₂ nanocrystallites.

Table 1: XRD analysis of SnO₂ nanocrystallites

2θ	(hkl)planes	Inter-planar distances(d)(nm)
26.6°	(110)	58.9
33.9°	(101)	55.3
38.0°	(200)	43.6
51.8°	(211)	59.3
57.9°	(002)	57.7
62.0°	(310)	49.3
66.0°	(301)	51.7
71.3°	(202)	47.5
78.7°	(321)	44.4

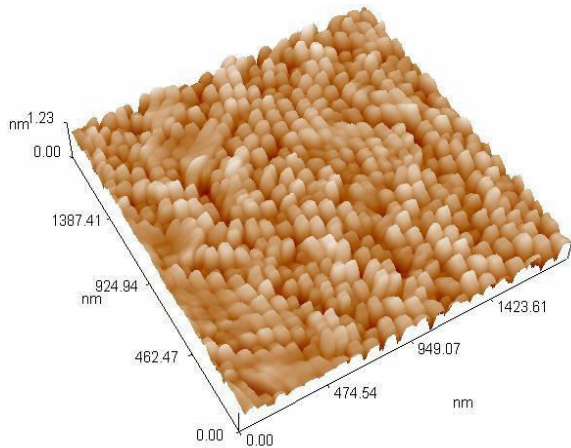


Fig. 2. SPM of SnO₂ nanoparticles

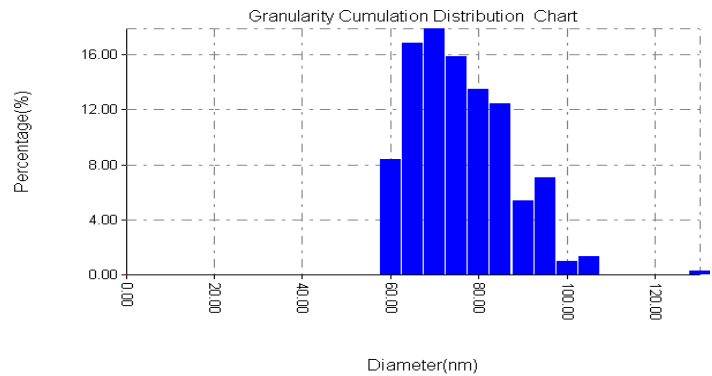


Fig.3. Granularity Cumulation distribution Chart.

B. Optical properties

The UV-VIS absorbance spectra of SnO₂ nanocrystallites are as represented in the Figure 4 and Figure 5 respectively. The characteristic absorption peak was found around 312 nm in the region ($275 \text{ nm} \leq \lambda \leq 350 \text{ nm}$). The substantial decrease in transmittance for SnO₂ nanocrystallites near the band edge indicates the better crystallinity and lower defect density for synthesized thin film. The SnO₂ thin film showed the transmittance of 78% in the spectral range of 350 nm – 800 nm.

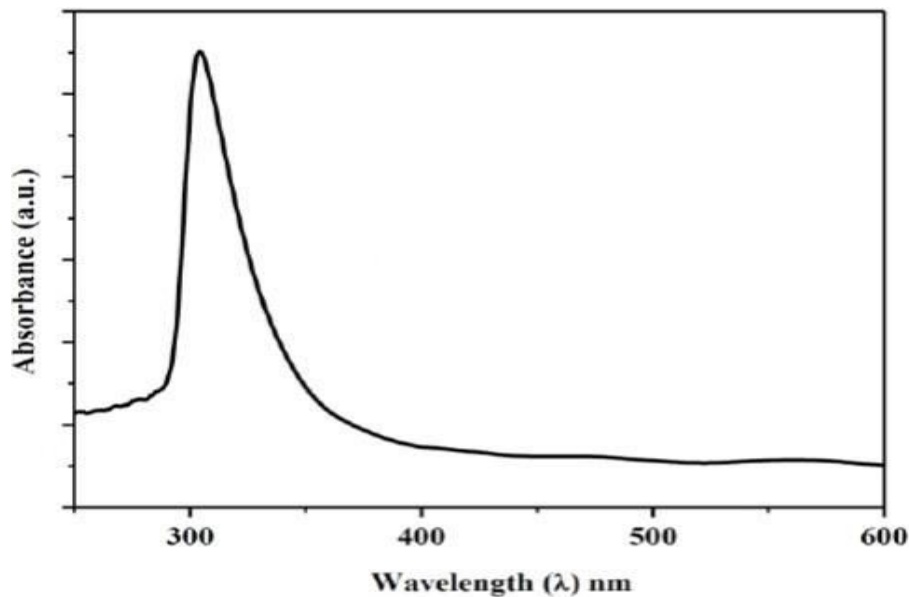


Fig. 4. The absorption spectrum of SnO₂

The optical band gap energy (E_g) of the semiconductor is calculated from Tauc relation [24]. A plot of $(\alpha h\nu)^2$ versus $h\nu$ shows intermediate linear region, the extrapolation of the linear part can be used to calculate the E_g from intersect with $h\nu$ axis as shown in Fig. 5. The resultant values of E_g for SnO₂ is found to be about 3.78 eV and 4.3 eV [25], The above two values may be related to the formation of nanostructures of SnO₂ and the bulk SnO₂, these values show a good agreement with the values published by other workers. [8,18]

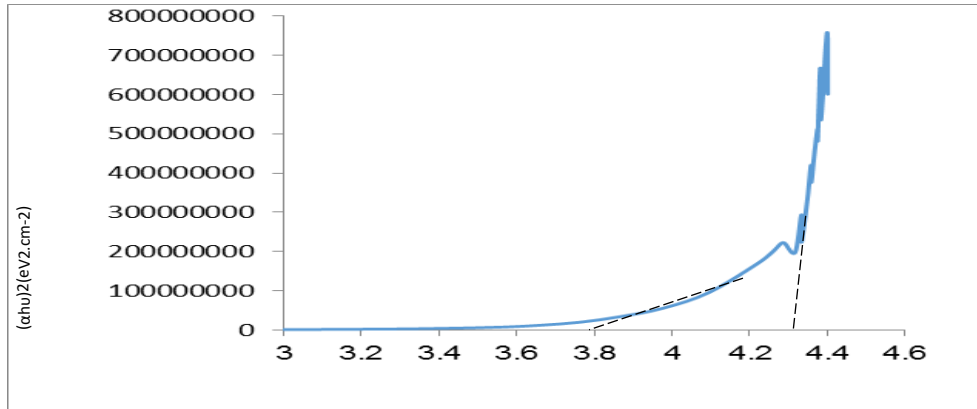


Fig.5. Plot of $(\alpha h\nu)^2$ vs. photon energy $(h\nu)$ for SnO₂.

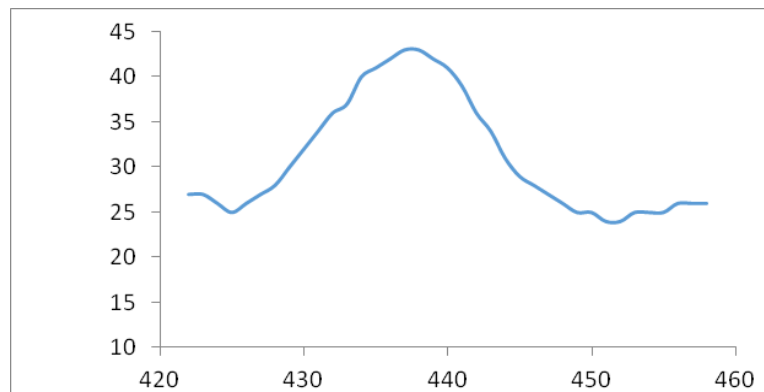


Fig.6. Photoluminescence emission spectra of SnO₂

Fig. 6 shows the photoluminescence emission spectra of SnO₂ nanoparticles at 280nm excitation, SnO₂ nanoparticles exhibit emission at 437nm. The emission maximum of 437 nm is lower than the band gap of the SnO₂ bulk, this peak can be attributed to the contribution of oxygen vacancies and defect in the SnO₂ nanoparticles [4,8].

IV. CONCLUSION

The chemical precipitation method was used to synthesized SnO₂ nanoparticles powder at 550 °C. The structural, morphological and optical properties of a SnO₂ sample were investigated. The XRD pattern is indexed to the tetragonal structure of SnO₂ and the calculated particle size in the range 8-10nm. The average particle size was 73.65nm of the SnO₂ nanoparticles estimated by SPM. The optical bandgap 3.78 eV of the SnO₂ were found and 4.3eV. The photoluminescence spectra of SnO₂ nanoparticles were at 312 nm excitation and exhibit emission in the spectral range of 350 nm – 800 nm.

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