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Effect of temperature on Structural and Optical properties of SnO_2 nanoparticles

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Abstract: *Tin oxide (SnO_2) nanoparticles were successfully synthesized via solvothermal method using the microwave oven technique at various calcination temperatures. The size of the nanoparticles was found to depend on calcination temperature. The near about spherical SnO_2 nanoparticles show a pure tetragonal rutile crystalline structure. A decrease in the band gap which is contrary to the quantum size effect was shown by the synthesized SnO_2 nanoparticles. SnO_2 nanoparticles were characterized by X-ray diffraction, Transmission Electron Spectroscopy, Fourier Transformed Infrared spectroscopy, UV-Visible Spectroscopy,*

Key words: *absorption, solvothermal, amorphous, oxidation.*

I. Introduction

Nanotechnology is the most essential and electrifying forefront field in Physics, Chemistry, Engineering and Biology. In semiconductor nanoparticles, the particle size influencesthe optical, catalytic, electric and magnetic properties [1]. Tin Oxide (SnO_2) is an n type semiconductor with direct band gap of 3.6eV of highly conducting, transparent and sensitive to gases [2-4]. SnO_2 nanoparticles used in gas sensors, solar cells, transistors, etc. It offers many technological applications such as oxidation of organics, solid state gas sensors, optical devices.Solvothermal method is good for the preparation of high quality oxide powder and the powder prepared has advantages of well defined grain[5], no aggregation, good dispersivity, moderate reaction conditions[6]. Our aim is to study the post-annealing temperature effect on nano-particle size, nano-structure and energy band gap of SnO_2 powders synthesized by the solvothermalmethod.

II. Experimental details

All reagents used were of analytical pure. Tin(II)Chloride dihydrate and Ureawere used as received. Ethylene glycol was used as solvent. Required amount of Tin(II)Chloride dehydrate and Urea were mixed in the solvent ethylene glycol. The solution so obtained was stirred to homogeneity using a magnetic stirrer for one hour and kept in a microwave oven bowl. A domestic microwave oven (M/O S20M WW-CG) operation at 2.45 GHz was used as the device. The microwave oven was operated at 1 min/cycle and cooled in between the cycles. Microwave heating was carried through until the solvent was dried up completely. The content was allowed to cool washed with double distilled water and acetone to remove any unwanted organic substances. The final product SnO_2 was taken out in the powder form. The sample was kept at different temperatures 100°C, 250°C, 400°C and 550°C in a muffle furnace.

III. Results And Discussion

3.1 Structural properties

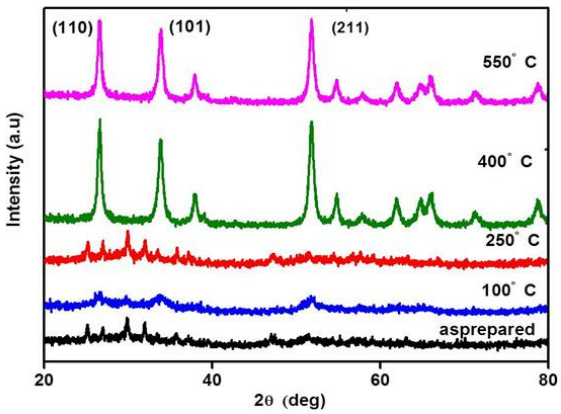

Figure 1 The XRD patterns of SnO_2 nanostructures at different annealing temperatures.

Fig. 1 shows the XRD patterns of SnO_2 nanostructures at different (unannealed, 100°C, 250°C, 400°C and 550°C) annealing temperatures. The number of reflection increases with increasing of the calcination temperature, but the degree of crystallinity does not change after 400°C representing fully crystalline SnO2 nanopowder.It is evidently seen that among all XRD patterns, the XRD pattern at 400°C shows better crystallinity and obviously broadened.The diffraction peaks located are in good agreement with SnO_2 tetragonal rutile structure (JCPDS file #770447). Peaks due to undesirable phases such as

not reacted SnO or Sn were not detected, thus pointing out the high purity of the product. Crystalline sizes in different directions are estimated in terms of Debye-Scherrer equation.

$$D = k\lambda / \beta \cos\theta \qquad (1)$$

where D is the crystalline size, K is a constant (0.9 assuming that the particles are spherical), λ is the wavelength of the incident X rays (0.1546nm), β is the full width at half maximum (FWHM), θ is the Bragg Diffraction angle. The crystallite sizes and the lattice parameters were calculated using XRDA software. Table 1 and table 2 shows thelattice parameters and particle size of the SnO_2 particle at 400°C and 550°C.The crystallite sizes of SnO_2 nanostructuresare 14.39nm and 15.09nm under400° and 550°C respectively. As seen in Table 1 and table 2, the width of the peaks decreases with increasing annealing temperature which refers to the growth of crystals size. The particle size decreases as the temperature increases from 400° to 550°C.

(hkl)	d (A°)	2 □(deg)	FWHM	Size (nm)
(110)	3.3487	26.598	0.49±0.011	16.62
(101)	2.6460	33.851	0.655±0.016	12.65
(211)	1.7639	51.786	0.631±0.012	13.90

Table 1 Lattice parameters and particle size of the SnO_2 particle at 400°C.

(hkl)	d (A°)	2 □(deg)	FWHM	Size (nm)
(110)	3.3496	26.590	0.465±0.011	17.55
(101)	2.6450	33.863	0.601±0.016	13.81
(211)	1.7635	51.802	0.634±0.015	13.92

TABLE 2 Lattice parameters and particle size of the SnO_2 particle at 550°C.

SEM Studies

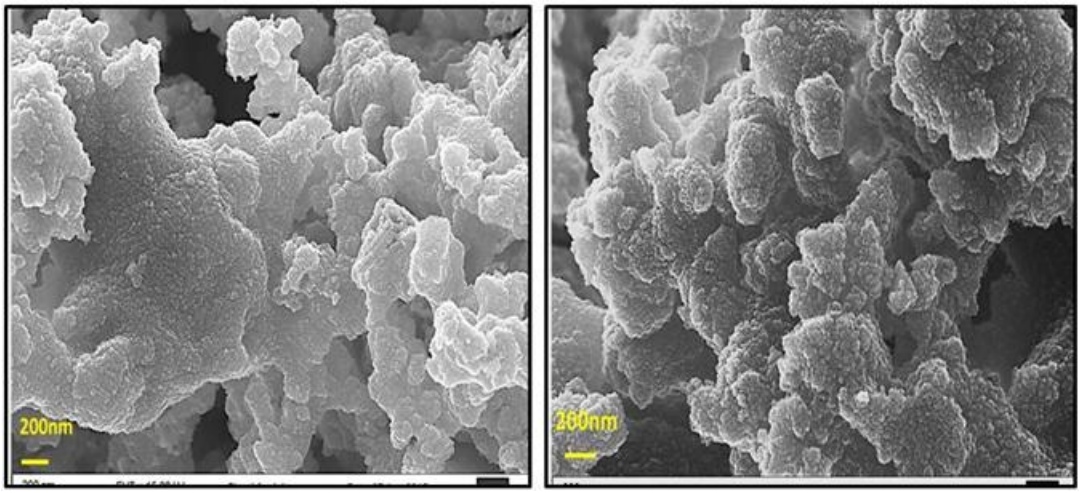

Figure 2 FESEM micrograph of SnO_2 nanostructure annealed at 400°C and 55

Fig. 2 shows FESEM micrographs of SnO_2 nanostructures annealed at 400° and 550°C respectively. All images indicate that the particles have non-uniform size with high degree of agglomeration.They are amassed into bigger particles parting some pores between which make them best for various uses.In addition, the changes in the morphology of SnO_2 nano particles strongly depends on the annealing temperatures. Both samples exhibit a spherical shape with a high degree of agglomeration among fine particles. Therefore, one cannot measure the particles size.The SEM examinations reveal that when the calcination temperature increases the agglomeration of non uniform particles increases.

TEM Studies

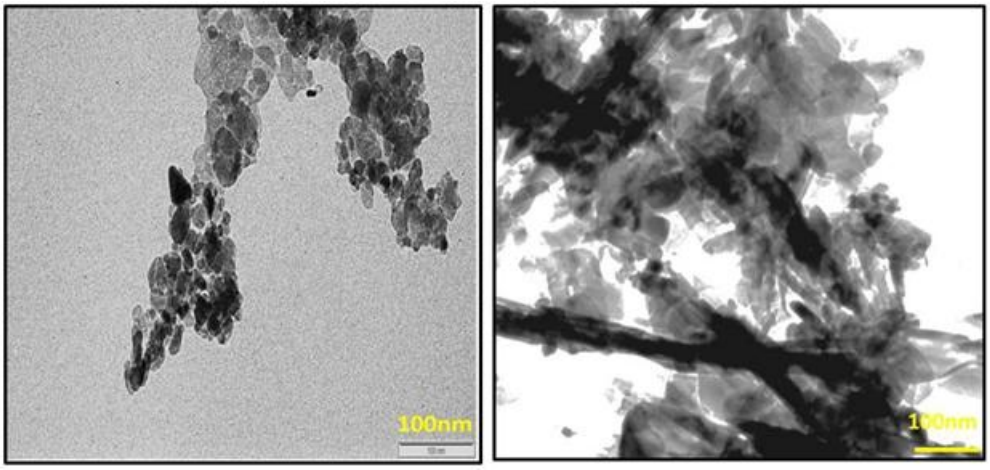

Figure 3 TEM image of SnO_2 nanostructure annealed at 400°Cand 550°C.

Fig. 3 shows TEM of SnO_2 nanostructures annealed at 400° and 550°C respectively.

Fourier Transform Infrared Spectroscopy (FTIR

It is an effective analytical instrument for detecting functional groups and characterizing covalent bonding information. Fig 4 shows FTIR spectra of SnO_2 nanostructures at 400° and 550°C. Fig. 6 shows very broad IR peak ranging from 3724cm^{-1} to 3025cm^{-1} is due to O-H stretching vibration and NH_3. For FTIR spectrum at 550°C, there is no peak at 2829cm^{-1}. The bands centered at 1610and 1354cm^{-1}might also related to water and NH_3. Increasing, the annealing temperature results in decreasing the depth of the bands around 1610 and 1354 cm^{-1}. An intense broad band at 630cm^{-1} attributed to the vibrations of Sn-O bond in SnO_2 [7]. Increasing the temperature results no change in its intensity.

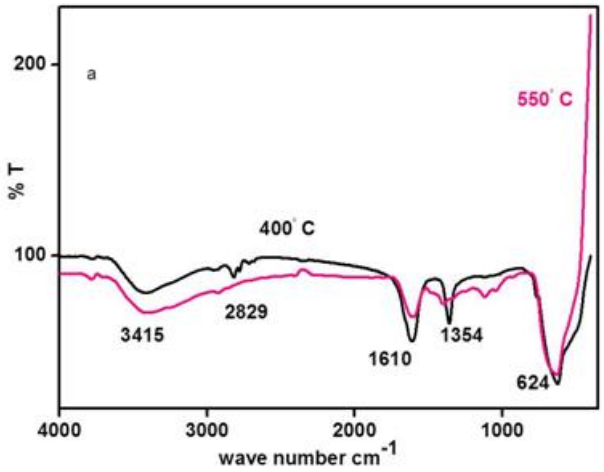

Figure 4 FTIR spectrum of SnO_2 nanostructure at 400° and 550°C.

3.2 Optical property

3.2.1. UV-Visible Spectroscopy

The optical absorption edges of the samples at 400°C and 550°C at 240 and 260nm. As SnO_2 is considered a direct band gap semiconductor, we use the Tauc relation

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

to estimate the optical band gap (Eg). A tauc plot can be drawn off $(\alpha h\nu)^2$ vs. $h\nu$. The point of extrapolation of the linear part that meets the abscissa will give the value of the band gap energy of the material. The Tauc's plot of the samples was shown in fig. 5.

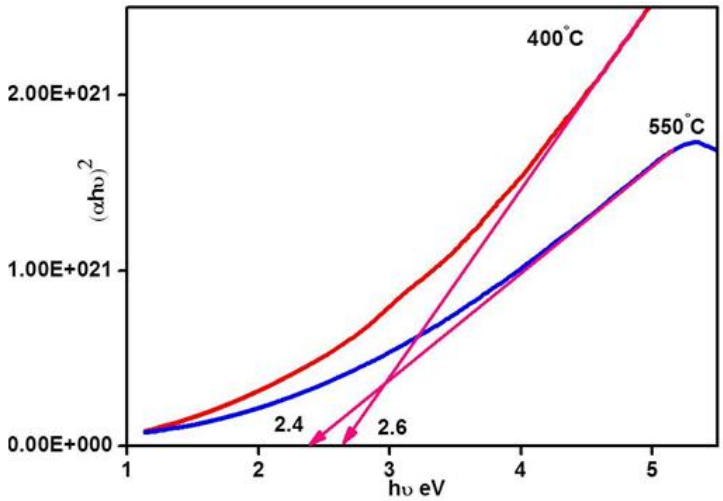

Figure 5 Band gap energies of SnO_2 nanostructures at 400^0C and 550^0C.

The intercept values on the energy axis have been found to be 2.6 and 2.4eV for 400°C and 550°C respectively. It can be found that the optical band gap energy is decreased when the temperature is increased. A decrease in the band gap which is contrary to the quantum size effect was shown by the synthesized SnO_2 nanoparticles.

IV. Conclusion

In conclusion, the successful synthesis of high purity, SnO_2 nanoparticles have been achieved via the solvothermal technique. The crystalline size was calculated from the Debye-Scherrer's formula. The crystallite size is calcination temperature dependent. The crystallite size of the samples increased from 14.39 to 15.09nm when the calcination temperature increases from 400 to 550°C respectively.The particle size decreases as the temperature increases. The SEM examinations reveal that when the calcination temperature increases the agglomeration of non uniform particles increases.The functional groups and characterizing covalent bonding information confirm SnO_2 nanoparticles. Optical studies have been carried out, using optical absorbance. The optical band gap energy is decreased when the temperature is increased due surface defecs.

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