Research Article

Crystal Structure, Lattice Strain, Morphology, and Electrical Properties of SnO₂ Nanoparticles Induced by Low Calcination Temperature

Panya Khaenamkaew¹,^{ID} Dhonluck Manop,¹ Chaileok Tanghengjaroen,¹
and Worasit Palakawong Na Ayuthaya²

¹Department of Basic Science and Physical Education, Faculty of Science at Si Racha, Kasetsart University, Sriracha Campus, Chonburi 20230, Thailand

²Department of Resources and Environment, Faculty of Science at Si Racha, Kasetsart University, Sriracha Campus, Chonburi 20230, Thailand

Correspondence should be addressed to Panya Khaenamkaew; pkheanumkhaw@yahoo.com

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The electrical properties of tin dioxide (SnO₂) nanoparticles induced by low calcination temperature were systematically investigated for gas sensing applications. The precipitation method was used to prepare SnO₂ powders, while the sol-gel method was adopted to prepare SnO₂ thin films at different calcination temperatures. The characterization was done by X-ray diffraction, scanning electron microscopy (SEM), and atomic force microscopy (AFM). The samples were perfectly matched with the rutile tetragonal structure. The average crystallite sizes of SnO₂ powders were 45 ± 2 , 50 ± 2 , 62 ± 2 , and 65 ± 2 nm at calcination temperatures of 300, 350, 400, and 450°C, respectively. SEM images and AFM topographies showed an increase in particle size and roughness with the rise in calcination temperature. The dielectric constant decreased with the increase in the frequency of the applied signals but increased on increasing calcination temperature. By using the UV-Vis spectrum, the direct energy bandgaps of SnO₂ thin films were found as 4.85, 4.80, 4.75, and 4.10 eV for 300, 350, 400, and 450°C, respectively. Low calcination temperature as 300°C allows smaller crystallite sizes and lower dielectric constants but increases the surface roughness of SnO₂, while lattice strain remains independent. Thus, low calcination temperatures of SnO₂ are promising for electronic devices like gas sensors.

1. Introduction

Tin dioxide (SnO₂) powders and thin films are essential for several applications such as a transparent electrode in panels [1], materials for rechargeable lithium batteries [2], solar cells [3], and gas sensing materials [4]. Due to the remarkable physical and chemical properties, SnO₂ has attracted much attention as a gas sensing material. The interactions between SnO₂ and neighboring gases (carbon monoxide/oxygen) enable this material as an excellent sensor [4].

The efficiency of the SnO₂ gas sensor depends on its particle size, connectivity of atoms, and characteristics of its

content. Researchers have shown that the execution of the SnO₂ sensor can be improved by reducing the particle sizes of SnO₂ in the nanometer length scale [4, 5]. The sensitivity increases rapidly when the particle size is less than 6 nm, considering as twice the thickness of the Schottky barrier [6]. The SnO₂ powder and thin films can be achieved by several methods, such as sol-gel process [6], chemical vapor deposition [7], a hydrothermal method [8], and sputtering methods [9]. To control the size of SnO₂ particles, there are at least two methods for succeeding. The powder synthesis method is the most common approach requiring standard chemistry apparatus and initial substances. The chemical interaction between reactant and reagent gives the desired

product, which appears as colloid or deposit in product solution. Varying concentrations of SnCl_4 and calcination temperatures can adjust the particle size [10], while other research works use spraying tin solution under pyrolysis or hydrolysis situation to obtain nanoparticles [11, 12]. The other method for deriving SnO_2 nanoparticle is a thin film growth process using various techniques such as dip coating or sol-gel coating [13, 14].

As mentioned in the previous paragraph, the efficiency of the SnO_2 sensor is firmly controlled by the crystal structure and particle sizes of SnO_2 . The precipitation method is a promising technique for controlling SnO_2 crystal structure and particle size because it produces high purity powder, nanoparticle size, and easy composition control and is of low cost [3–5, 15]. However, only a few studies have examined crystal structure at low calcination temperature, morphology, and electrical properties in SnO_2 powder. Earlier studies reported the features above 400°C calcinations [6, 15–17]. The physical and mechanical properties of SnO_2 powders may vary with temperatures. Hence, before using nano- SnO_2 powders, it is crucial to check the above features at low-temperature conditions. Naseem et al. [18] first reported the lattice strain of SnO_2 powder, which can vary due to the solvents used in the synthesis process. [18]. Furthermore, there is no report in the literature about the lattice strain generated between the crystal structure or the lattice mismatch during the phase transformation process. This lattice strain usually produces the internal electric, which helps in sensing behavior due to the variations in energy bandgap. However, sufficient research is required to strengthen the above statement. The main challenge is how to use the nano- SnO_2 powder and thin films as a gas sensor in industrial uses.

In the present work, we have examined various characteristics of SnO_2 thin films and powders by varying temperature. The crystallite structure, that is, particle size, lattice parameter and lattice strain, morphology, topography and roughness, optical properties, and dielectric property, was explored for achieving a suitable condition to obtain various morphologies of SnO_2 nanoparticle for the application of devices in sensors.

2. Materials and Methods

2.1. SnO_2 Powder. So far, various synthesis methods are being used for preparing SnO_2 powders [19, 20]. Here, we have used the precipitation technique to obtain SnO_2 nanoparticles [17], and tin tetrachloride pentahydrate ($\text{SnCl}_4 \cdot 5\text{H}_2\text{O}$) was used as starting materials in the precipitation process. $\text{SnCl}_4 \cdot 5\text{H}_2\text{O}$ ($M = 260.7 \text{ g/mol}$ MERCK, KGaA, Germany) and NH_4OH (25% conc., 35 g/mol, from MERCK, Germany) were used as precursors. First, 26.07 g of $\text{SnCl}_4 \cdot 5\text{H}_2\text{O}$ was dissolved in (gently dropped into) distilled water. Second, 14 g of NH_4OH was added dropwise into the distilled water. Then, NH_4OH solution was added to $\text{SnCl}_4 \cdot 5\text{H}_2\text{O}$ till the pH becomes 9.0, precipitating milky white $\text{Sn}(\text{OH})_4$. Subsequently, the $\text{Sn}(\text{OH})_4$ solution was filtered, washed, and dried in an oven at 110°C for 24 hours. The reactions may be written as follows:

The calcination temperature is an important parameter for determining the electrical property of a substance. Hence, SnO_2 powder was made in a round-shaped plate by a single axis hydraulic press at $20.5 \pm 0.5 \text{ MPa}$ pressure. Then, the samples were set into a tubular furnace by varying the calcination temperatures as 300, 350, 400, and 450°C for three hours with a proportion of 3°C/min heating. The density was calculated using weight and thickness. Figure 1 presents the preparation of SnO_2 nanoparticles.

2.2. SnO_2 Thin Film. Tin-dichloride-dihydrate ($\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$) was first dissolved in isopropanol, and then deionized water was added to it at a ratio of 0.6 : 6 : 0.4. While adding the deionized water, continuous stirring is required to start the hydrolysis and polycondensation reactions. Besides, 0.05 ml of acetic acid was used as a catalyst. Then, the magnetic stirrer was used for 3 h at 25°C to make the sol completely homogeneous and transparent, as reported by Terrier et al. [21]. The ITO-coated glass substrate [13], which is available in the market, was cut into equal pieces with a similar dimension of $1 \times 1 \text{ inch}^2$. The substrates were washed with deionized water and acetone to remove dirt. To overcome further impurities attached to the ITO substrate, an ultrasonicator was used to rinse with methanol and deionized water alternatively thrice. Substrates were then immersed into an earlier prepared solution for 5 min and pulled out with a fixed velocity with 3.0 mm/min, and the same procedure repeated ten times to achieve a desired thickness of the film.

2.3. Characterization. The crystal structures were obtained by X-ray diffraction (XRD, Bruker D8 Advance with Eulerian Cradle; $\text{CuK}\alpha 1$ with $\lambda = 1.54060 \text{ \AA}$, Spain) within the diffraction range of $2\theta = 20^\circ$ to 80° [7, 8]. The diffraction data were recorded at a 3° min^{-1} scan rate for the analyses of crystal phases [22]. The crystalline size ($\beta_{\text{crystalline}}$) and lattice strain of the sample ($\beta_{\text{strain}})_n$ are related to the width (β_r) [23]:

$$\beta_r = \beta_{\text{crystallite}} + \beta_{\text{strain}}. \quad (3)$$

Here, ($\beta_{\text{crystalline}}$) values were estimated from the “Scherrer–Gottingen equation.” ($\beta_{\text{crystalline}}$) can be calculated by considering Bragg reflections with diffraction angle (2θ) [24]:

$$\beta_{\text{crystallite}} = \frac{K\lambda}{L \cos \theta}, \quad (4)$$

where K is a constant and considered as dimensionless shape factor ($K = 0.89$); λ is a wavelength of $\text{CuK}\alpha$ radiation. Hence, the lattice strain can be represented as follows:

$$\beta_{\text{strain}} = \eta \tan \theta, \quad (5)$$

where η denotes the strain of the sample. Thus, the total width of the diffraction peak becomes

FIGURE 1: The precipitation process of investigated SnO_2 powder in this experiment.

$$\beta_r = \frac{K\lambda}{L \cos \theta} + \beta_{\text{strain}}, \quad (6)$$

$$\beta_r \cos \theta = \frac{K\lambda}{L} + \eta \sin \theta. \quad (7)$$

In the graph, $\beta_r \cos \theta$ against $\sin \theta$, one can obtain the lattice strain of the sample η from the slope and the crystallite size (L) from the intercept ($K\lambda/L$).

Scanning electron microscopy (SEM, Hitachi S-3400N, Japan) with an energy-dispersive X-ray spectrometer (EDX, Oxford instrument, UK) was used to investigate the arrangement of atoms in the crystal, particle size, and chemical compositions of SnO_2 powders. Gold and carbon were coated onto samples for SEM and EDX analysis, respectively.

The topography and crystallite size of SnO_2 powders were investigated by atomic force microscopy (AFM, Nanosurf, Nanosurf Easy Scan 2, Switzerland) at room temperature. The principle and experimental setup of the AFM is described in the literature [25]. A commercial tip cantilever system (Scout 350, Nunano, UK) with spring constant of $k = 42 \text{ N/m}$ and tip apex radius less than 10 nm was used. Measurements were carried out in noncontact mode acquiring $256 \text{ pixels} \times 256 \text{ pixels}$ images with a rate of scanning 2 to 5 Hz .

The permittivity was calculated from the measured capacitance by using a parallel plate method. The vector components of capacitance (C) and dissipation (D) were measured by the instrument. Finally, a computer program was used to calculate the relative permittivity and tangential loss as described from the following equation [26]:

$$\epsilon' = \frac{tC_p}{A\epsilon_0}, \quad (8)$$

$$\epsilon'' = \frac{t}{\omega R_p A \epsilon_0}. \quad (9)$$

Here, t denotes the thickness and A signifies the area of SnO_2 samples.

A double beam UV-Vis Spectrophotometer (Biochrom Libra S80, UK) was used to perform the optical properties of SnO_2 films at room temperature with wavelength range $200\text{--}800 \text{ nm}$. From the reflection $R(\lambda)$ and transmission $T(\lambda)$ data, the absorption coefficient (α) was calculated using the following equation [27]:

$$\alpha(\lambda) = \frac{1}{t} \ln \left[\frac{(1 - R(\lambda))^2}{T(\lambda)} \right]. \quad (10)$$

Here, t is the thickness of the film. For direct energy bandgap, the above equation can be written as

$$\alpha \approx A \sqrt{(h\nu - E_g)}, \quad (11)$$

where E_g is the optical bandgap and $h\nu$ is the energy of a photon. A plot was generated earlier $(\alpha h\nu)^2$ versus $h\nu$ for films at different calcination temperatures [28].

3. Results

3.1. Effect of Calcination Temperature on the Crystal Structure of SnO_2 Powder. At low calcination temperature, the measured XRD pattern of the product exhibited a very rough signal as several stray peaks appeared over the scan angles. However, some important broad peaks of SnO_2 were strong enough to be identified. Figure 2 presents the XRD patterns obtained from the powder SnO_2 samples at various calcined temperatures. The sharpness and an increase in peak intensity are attributed to the rise in calcination temperatures by crystallinity [16, 17]. The strong intensity was observed along (110) plane, while comparatively weak intensity was detected along (101) and (211) planes. However, the XRD results of calcination temperature of 400°C and 450°C appeared more crystalline

FIGURE 2: The XRD patterns of SnO_2 powder calcined at different temperatures.

than 300°C and 350°C. The crystallinity of the 300°C regimes indicated an amorphous structure as low intensity and broad peak attribution were found. Since the calcination temperature was increased, the intensity and width of XRD peaks became high and narrow, respectively, suggesting that powder SnO_2 obtained enough energy to form a perfect crystalline form.

Using equation (7) and Figures 3(a)–3(d), the average sizes of nanocrystalline SnO_2 powder were obtained, which could be 45 ± 2 , 50 ± 2 , 62 ± 2 , and 65 ± 2 nm. The data in Table 1 manifest a linear relationship between $\beta_r \cos \theta$ and $\sin \theta$. From the experiments, the corresponding lattice strains became 0.0087, 0.0102, 0.0116, and 0.0084 while treated at 300, 350, 400, and 450°C, respectively. Table 1 presents the crystallite size of the sample, which was increased by the rise in calcination temperature. Hence, the continuing growth process was also found in the present research, as well as in the literature [10, 11]. Besides, a rise of the crystalline size of about 5 nm was observed after calcination at 400°C for 3 hours, which explains a small growth trend under such conditions. The XRD data revealed the polycrystalline nature of SnO_2 powder, which was fitting well in the tetragonal crystal with a space group $P42/mnm$. The corresponding lattice parameters were found as $a = b = 4.73 \pm 0.04$ Å, $c = 3.18 \pm 0.04$ Å [2]. The lattice strains were 0.0102 and 0.0116, with 350°C and 450°C, respectively. The strength increases with the rise in temperature, which signifies that the structure might reorient due to the process of phase transformation. The internal electric field was produced between the grains of SnO_2 powder and enhanced the electrical property of the SnO_2 gas sensor.

3.2. Effect of Temperature Calcinations on the Morphology of SnO_2 Powder. Figure 4 depicts the SEM micrographs of SnO_2 powder calcined at different temperatures. At low temperature (300°C), the distribution of SnO_2 particles is inhomogeneous, as shown in Figure 4(a), while raising the temperature (400°C) causes an improvement in particle

distribution. Besides, Figures 4(b)–4(c) reveal that the crystallite sizes of each sample are uniform, depending on the calcination temperature. It is to be noted that a low calcination temperature takes more energy for crystal growth than high calcination temperature, as reported in the literature [12, 13, 29]. However, the SEM images show that high calcination temperature (400°C and 450°C) helps to form more crystalline states than the lower one (300°C and 350°C); this finding matches with the XRD result [6]. Figure 5 shows various crystallite sizes observed in the calcination temperature of 300°C and 350°C. These approximations obtained from the SEM images and the crystallite sizes within the range of 30 to 60 nm show an excellent agreement to the data presented in Table 1.

The aggregation of the 200–400 nm particles only occurred in the samples prepared at the lower annealing temperature. These nonhomogeneous particles are usually observed when the annealing temperature of a sample was decreased to 300°C–350°C [30]. This is because the lower annealing temperature needs more energy growth than a higher annealing temperature. According to the fact that the SnO_2 are crystallizes into the polymorphs, the observed 200–400 nm particles of SnO_2 (Figure 5(b)) are composed of rutile type ($P42/mnm$) and pyrite type ($Pa3$) phases.

The purification of SnO_2 powder investigated by the EDX indicated the atomic weight ratio of Sn by the increase in calcination temperature. At 300°C, the chloride atom left from the precipitation process and dominated over the synthesized crystal (Figures 6(a)). However, the Sn atom was found as a larger weight percentage in other temperatures (Figures 6(b)), as shown in Table 2.

Figure 7 depicts a series of AFM topographs of SnO_2 powder with different calcination temperatures. The surface roughness and the crystallite size (40–100 nm) increase by rising calcination temperatures as the atoms obtain enough energy at higher temperatures. There is a probability of recrystallization at higher temperatures as atoms often change their position. This finding is similar to the results reported in the literature [31, 32]. The density and

FIGURE 3: Plot of $\beta_r \cos \theta$ against $\sin \theta$ of SnO_2 calcined at (a) 300°C, (b) 350°C, (c) 400°C, and (d) 450°C extracted from the XRD patterns.

TABLE 1: Structural parameters of SnO_2 powder, lattice: tetragonal S.G. = P42/mmm (136).

Calcine temp. (°C)	Slope (η)	Intercept ($K\lambda/L$)	L (nm)	a (Å)	c (Å)
300	0.0087	0.0030	45.70	4.7648	3.1836
350	0.0102	0.0027	50.78	4.7382	3.1871
400	0.0116	0.0022	62.32	4.7397	3.1877
450	0.0084	0.0021	65.29	4.7387	3.1865

homogeneity of the SnO_2 powders were improved after calcination, showing a strong agreement with the results obtained from XRD. Not only does calcination induce an apparent increase in the size of crystal but also it changes the phase content, which is the ratio between rutile (P42/mmm) and pyrite (Pa3) type phases. However, at a low calcination temperature as low as 300°C–450°C, the phase ratio was evaluated from the diffractograms and found to be quite stable (the results not shown here).

The size of the crystals increases by increasing the temperature of calcination. However, it is recommended to have a small particle size for gas sensors as a higher surface-to-bulk volume ratio increases the adsorption of gases at the sensor surfaces, leading to enhanced sensitivity. The results shown in Figures 2–7 indicate that the crystallite size of SnO_2 powders is improved in the range of 40–100 nm after calcination. Hence, the present samples are promising for sensor applications.

3.3. Effect of Temperature Calcinations on the Dielectric Constants of SnO_2 Powder. The electrical features of SnO_2 powders were investigated by measuring the dielectric

constant at different calcination temperatures. Figure 8 presents the behavior of dielectric properties with the effect of frequency at normal temperatures. The dielectric measurement of nanoparticles is a difficult task, and the present results could influence the apparent porosity. Pressing the SnO_2 samples at 30.5 ± 0.5 MPa could eliminate the porosity effect of the dielectric measurement. During the analysis, several LC resonances were noticed on the graphs between dielectric constant and frequency. The resonance might come from the inductance of the wires but was removed before calculation.

The dielectric constant (ϵ') decreases abruptly at lower frequencies while achieving a saturation at frequencies above 10 MHz. The inset of Figure 8 shows the saturated ϵ' at high frequencies, illustrating ϵ' as almost frequency independent and can be explained by the polarization mechanism. The dipole moment changes by varying applied electric field at low frequency, resulting in polarization. But dipole moment did not follow specifically by changing the applied electric field, resulting in a decrease in ϵ' at a higher frequency. The values of ϵ' obtained in the present research are much lower than the reported values [23, 29], attributing to the inhomogeneity of the dielectric structure. The inhomogeneity could occur due to the presence of

FIGURE 4: The SEM images of investigated SnO_2 powder calcined under (a) 300°C, (b) 350°C, (c) 400°C, and (d) 450°C.

FIGURE 5: Crystallite size approximation obtained from high magnification SEM images of annealed under (a) 300°C and (b) 350°C of SnO_2 sample.

FIGURE 6: EDX spectra data for SnO_2 at calcination temperature of (a) 300°C and (b) 400°C.

TABLE 2: The atomic and weight percentages of the mixture of Sn and oxide obtained from the EDX technique.

Calcinations temperature (°C)	Sn element		O element	
	Weight (%)	Atomic (%)	Weight (%)	Atomic (%)
300	21.78	3.21	—	—
350	75.97	34.76	17.19	58.34
400	78.54	41.26	13.00	50.66
450	74.33	26.46	18.30	48.33

FIGURE 7: AFM topographies with 3D structure of SnO_2 films: (a) 350°C ($5 \times 5 \mu\text{m}^2$) and (b) 450°C ($2.5 \times 2.5 \mu\text{m}^2$) of calcination temperatures.

grain structure, porosity, crystal defects, and stoichiometry. Figure 9 presents the plot of dielectric losses (ϵ'') at different calcination temperatures and frequencies. At higher frequency, ϵ'' decreases and, thus, polarization occur. At 400 Hz, ϵ'' becomes maximum, indicating the dissipation of samples and forms like loose materials while using in an external electric field.

3.4. Effect of Temperature on the Optical Bandgap of SnO_2 Films. Figure 10 presents an optical absorbance of SnO_2 nanoparticles at different temperatures of calcination. In the spectra, absorption is high at the lower wavelength region,

attributing two sets of wavelengths. A sharp increase in absorption manifests in the first region ($200 \text{ nm} \leq \lambda \leq 250 \text{ nm}$), while, in the second region ($250 \text{ nm} \leq \lambda \leq 290 \text{ nm}$), an abrupt fall in absorption occurs. The average absorbance in the visible region lies within 25–30% by varying the calcination temperatures, while maximum absorbance comes to 38.5% at 450°C . By reducing the calcination temperature, the UV-visible absorbance shifts towards a smaller wavelength due to the decrease in particle size. Furthermore, the smaller sizes of particles can absorb at shorter wavelengths, and, thereby, the bandgap increases, showing a good agreement with the present SEM and XRD data.

FIGURE 8: Dielectric properties of SnO_2 powders with different calcination temperatures as a function of driven frequencies.

FIGURE 9: The dielectric loss of SnO_2 powders with different calcination temperatures as a function of driven frequencies.

FIGURE 10: The absorption of SnO_2 films with different calcination temperatures.

FIGURE 11: The energy bandgaps of SnO₂ films with different calcination temperatures (Touc plot of $(\alpha h\nu)^2$ with photon energy in SnO₂ thin film).

Figure 11 presents the plot of energy of the optical bandgap at different calcination temperatures. The direct bandgap energy (E_d) values can be found from the intercepts with the energy axis. The bandgaps are 4.85, 4.80, 4.75, and 4.10 eV for 300°C, 350°C, 400°C, and 450°C, respectively. Notably, E_d values calculated from the present analysis are higher than those found in the reports [5, 16, 18]. These larger values are achieved due to the variation of the stoichiometry of the concentration of point defects associated with SnO₂ films. Furthermore, E_d decreases with the increase in crystallite size, although independent of the lattice strain. A similar trend has been observed in earlier reports [33].

4. Conclusions

We successfully prepared nano-SnO₂ particles by the precipitated method. XRD analysis revealed a tetragonal rutile structure with P42/mnm (136) phase group from the synthesized compounds. The average crystalline size increases from 45 ± 2 , 50 ± 2 , 62 ± 2 to 65 ± 2 nm due to the increase in calcination temperature from 300, 350, 400, to 450°C, respectively. These results also show the persistence of lattice strain, which is independent of calcination temperature. SEM images show that the average particle size and grain size increased while increasing the calcination temperature. EDX analysis of the samples showed the existence of tin and oxygen in the pure sample. AFM images showed that the rise in calcination temperatures contributes to high surface roughness and large crystallite size. The dielectric constant was much lower than the expected value. The energy bandgap of SnO₂ thin films was independent of their lattice strain but decreased with an increase in their crystallite size. Lattice strain was ambiguous and needed further investigation. However, the morphology of SnO₂ powder can be altered in terms of particle size and crystal structure by different calcination temperatures, which could improve the sensitivity of the sensor. Hence, the present research caused by shallow calcination temperature should be carried out for future direction.

Data Availability

The data used to support the finding of this study are included within the article.

Conflicts of Interest

The authors declare no conflicts of interest.

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