

# Preparation of tin oxide (SnO<sub>2</sub>) thin films using thermal oxidation

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**Abstract.** The present research study deals with the preparation of tin oxide (SnO<sub>2</sub>) nanostructures using thermal oxidation method. At first, Sn thin film was deposited on silicon (Si) substrate by thermal evaporation and then, thermal oxidation of the deposited Sn thin film was carried out at the growth temperature of 100°C with growth time of 1 hour in tube furnace. The structural property of SnO<sub>2</sub> nanostructures was investigated by using FESEM and EDX. The FESEM results showed that Sn was successfully grown on Si substrate and the SnO<sub>2</sub> nanoparticles with diameters of 97.5nm to 142nm were recorded. It was observed that the particles were agglomerated to form the SnO<sub>2</sub> particles. The radiation of sunlight illumination was conducted for four consecutive sunny days and the results showed that the highest reading 189.9 W/m<sup>2</sup> was recorded at day two for the daytime temperature 38°C. It was also noticed that the highest solar radiation percentage at day two was measured 18.9%.

## 1. Introduction

Tin Oxide (SnO<sub>2</sub>) thin films are one of the nanostructures semiconducting oxide materials that are being received broad attention due to unique electrical, physical, chemical, structural and optical properties. It is well known that n-type semiconductors with a wide band gap of 3.6-3.8 eV can be used in various fields, such as a window layer in solar cells, heat reflectors in solar cells, various gas sensors, transparent conducting electrodes, photochemical and photoconductive devices in liquid crystal display, gas discharge display, lithium-ion batteries etc. [1-6]. The properties of tin oxide for the performance of the sodium-ion batteries were investigated [7]. Tin oxide thin films are the most attractive films that are used in automotive sector as an anticorrosive surface treatment of a carbonaceous bipolar plate in proton exchange membrane fuel cells [8]. Using a sol-gel process, nanostructured tin oxide powders were prepared successfully [9]. The obtained results showed that crystallization of tin oxide started at about 250°C and the growth continued at higher temperatures as the organics were removed. On the other hand, it was found that nanocrystalline tin oxide powders were formed only after the hydroxyl groups were completely removed at nearly 600°C.

Mixed semiconductor oxide nanostructures of tin dioxide/tin monoxide (SnO<sub>2</sub>/SnO) have been produced using hydrothermal method [10]. The morphological, structural and photoluminescence properties of the fabricated nanostructures were investigated. For the growth of tin oxide nanostructure, silicon wafer was used as a substrate and using thermal evaporation method, tin oxide nanowires have been synthesized for gas sensing application [11]. The synthesized tin oxide nanostructures were characterized using XRD, EDX, SEM and TEM. In this investigation, vapor-liquid-solid (VLS) growth of the tin oxide nanowires was confirmed.



Tin oxide thin films have been synthesized using spray pyrolysis and electron beam evaporation methods [12]. The structural, electrical and optical properties were investigated under different operating conditions of solution flow rate, substrate temperature and deposition rate. The obtained results revealed that conductivity of the deposited films was dependent on structure of films and substrate temperature. The doping effect of neodymium (Nd) on the characteristics properties of tin oxide thin films was investigated [13]. The optical, electrical, crystallographic and morphological characterizations revealed that neodymium doping significantly influenced the properties of tin oxide thin films. Due to the high transparency for visible light and good electrical conductivity, tin oxide thin films are used for numerous applications, such as low-emissivity coatings for glass windows and solar cells [14]. Each application needs different properties of the tin oxide thin films. These properties can be varied by adjusting different parameters involved in the chemical vapour deposition (CVD) process by which tin oxide layer can be deposited on the substrate. The growth of tin oxide nanomaterial was carried out and the optimum growth parameters by varying the growth temperature and growth time were investigated [15]. The obtained results showed that samples were grown on silicon substrate and exhibited like nanobelts and nanorods. Moreover, tin oxide nanomaterial displayed fluorescence and photoluminescence signals.

In this research study, SnO<sub>2</sub> nanostructures were prepared on Si substrate following a route of thermal evaporation and then, thermal oxidation. The properties of these SnO<sub>2</sub> nanostructures have been studied using Field Emission Scanning Electron Microscope (FESEM) with Energy Dispersive X-Ray (EDX) analysis. The efficiency or performance of these fabricated SnO<sub>2</sub> samples was also measured in this study.

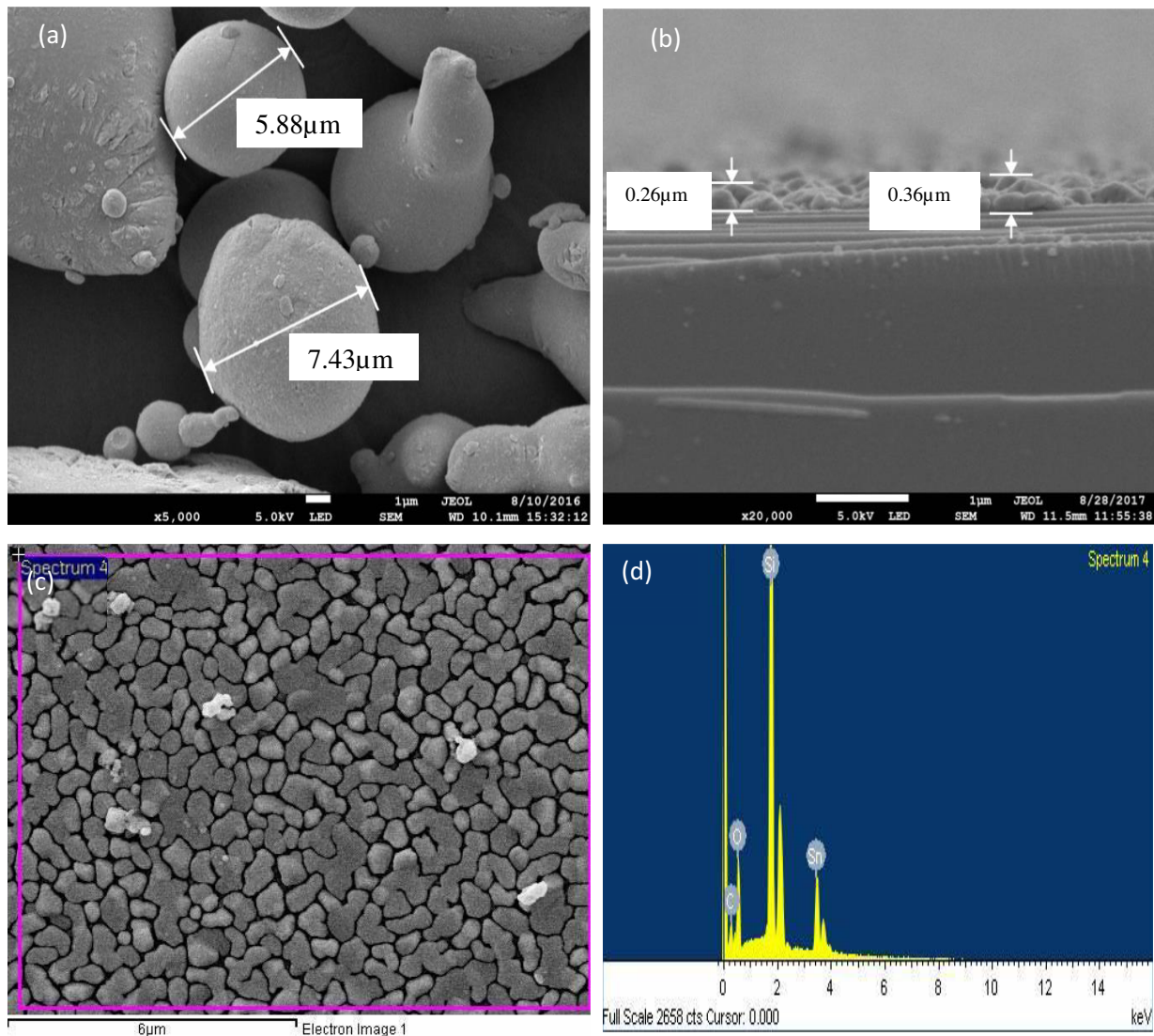
## 2. Experimental

Silicon (Si) wafer p-type (100) with dimension 1.5cm x 2.5cm and thickness of 500±50 µm was used as a substrate for the growth of SnO<sub>2</sub> nanostructures. At first, the Si substrate was cleaned using freshly prepared Piranha solution to remove dust and organic mixture or contaminants. A mixture solution of sulphuric acid (H<sub>2</sub>SO<sub>4</sub>) and hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) (3 : 1) was prepared and the cleaning process was conducted using ultrasonic vibrations for 30 minutes. After that, the Si substrate was rinsed with distilled water and dried with air gun. The tin powder (Sn) was weighed at 0.05g which was then deposited on the Si substrates in thermal evaporator. The base pressure in thermal evaporator was set at 1.60x10<sup>-4</sup> Pa with target-substrate distance of 10.5mm at room temperature, voltage 0.9V and current 20-25A. The deposition time was constant for 30 minutes for each sample. The obtained Sn thin film sample was characterized by using FESEM and EDX analysis. Next, the thermal oxidation was carried out by using tube furnace with the oxidation temperature at 100°C. The heating rate used was 5°C/min and the oxidation time is 1h. Finally, the substrate was cool down to the temperature and then unloaded from the furnace. The morphological studies were carried out by the FESEM JEOL, JSM-7800F with EDX analysis. The obtained SnO<sub>2</sub> sample was also tested for radiation of sunlight illumination by solar power meter.

## 3. Results and Discussion

### 3.1. FESEM and EDX

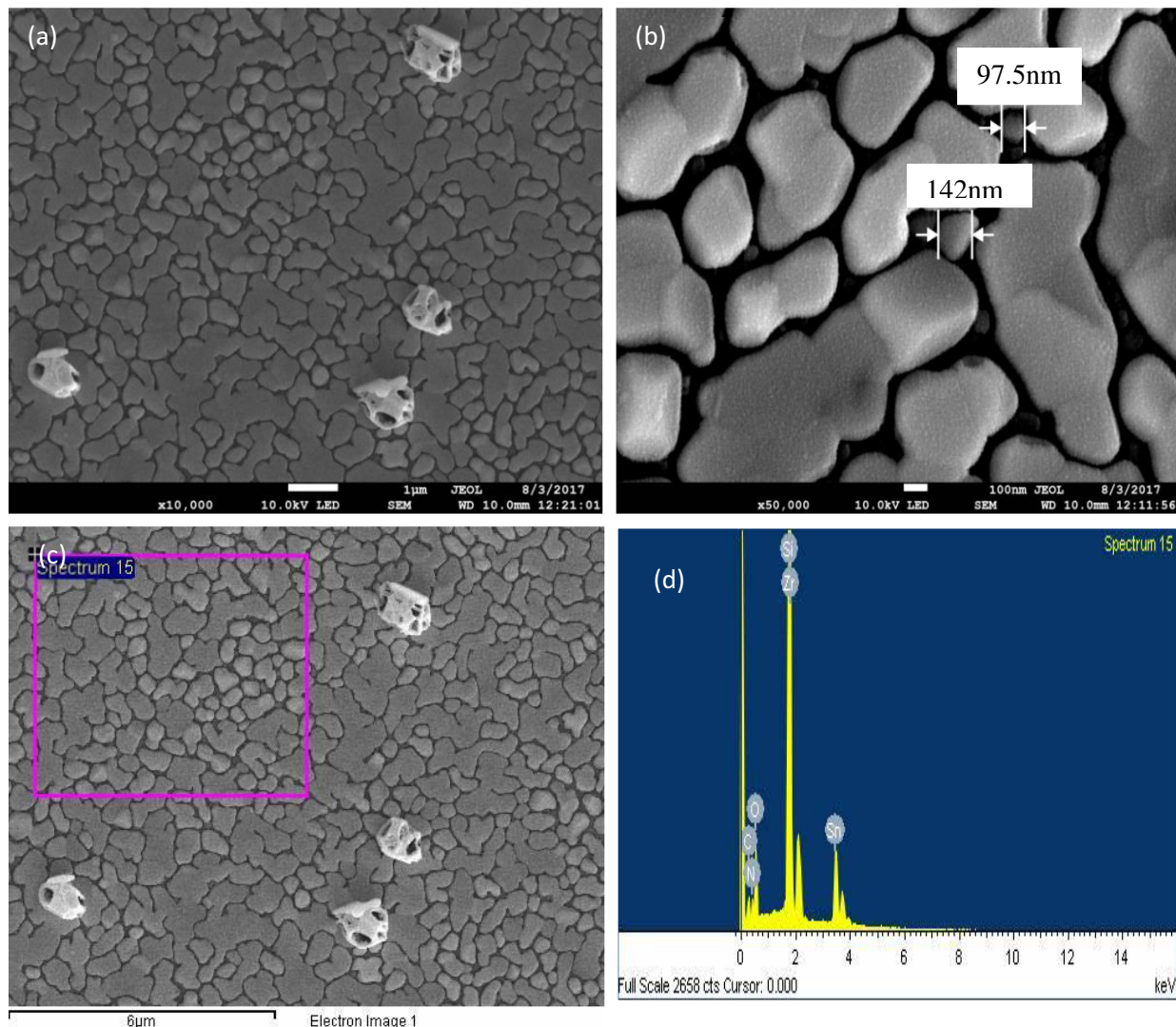
Figures 1(a)-(d) show the characterization results using FESEM and EDX. Figure 1(a) shows the average particle size of the Sn thin film using 5000x magnification. The average particle size varies from 5.88µm to 7.43µm with some of the particles were almost round shape, and other particles were irregular shapes. The obtained results also showed that the sizes of the particles are not uniform which means that the evaporation process was not uniformly distributed over the surface. It suggests that tin was not uniformly evaporated may be because of the Si substrate had a rough surface during the cleaning process. There might be a possibility that the evaporated material attacked the substrate mostly from a single direction. Figure 1(b) shows the cross section of the Sn films. The obtained results showed that thickness of the deposited Sn film varies from around 0.26 µm to 0.36 µm.



**Figure 1.** FESEM image of Sn thin film after thermal evaporation (a) the average particle size of Sn and (b) cross section of Sn layer on the Si substrate (c) EDX point surface of Sn layer on Si and (d) EDX spectrum of Sn thin film.

Figures 1(c)-(d) show the EDX results of the Sn film and the elements consist of Si, C, O and Sn meaning that Sn was successfully grown on the Si substrate. Figures 2(a)-(d) show the FESEM and EDX image of SnO<sub>2</sub> at growth temperature 100°C. Figure 2(a) shows the FESEM image of the surface of SnO<sub>2</sub> thin film using x10000 magnification. Figure 2(b) clearly shows that agglomeration of the particles occurred with irregular shapes of tin oxide and the average diameter varies from 97.5nm to 142nm. The tin film was not fully oxidized at this temperature due to the melting point of tin is 232°C. It is believed that grain rotation induced grain coalescence mechanism appears in this growth of the SnO<sub>2</sub> crystals due to the appearance of spherically elongated crystals [16]. Figures 2(c)-(d) show the EDX result of SnO<sub>2</sub> at growth temperature 100°C that confirms the elements consist of Si, Sn, C, and O. The particles sizes were reduced to about 80% after thermal oxidation of Sn thin film.



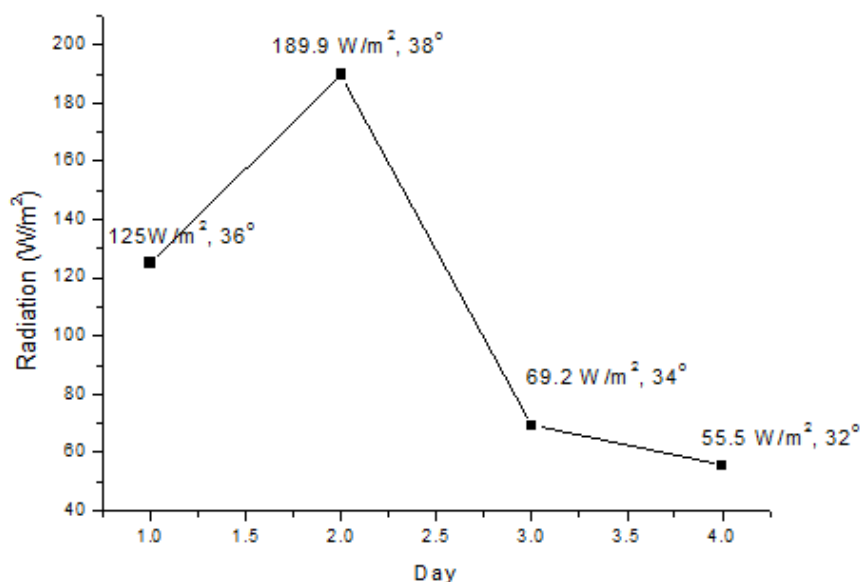


**Figure 2.** FESEM image of  $\text{SnO}_2$  at  $100^\circ\text{C}$  after thermal oxidation (a) surface of  $\text{SnO}_2$ , (b) grains size of  $\text{SnO}_2$  particles, (c) EDX point surface of  $\text{SnO}_2$  (d) EDX of  $\text{SnO}_2$ .

### 3.2 Solar Power Meter (Sunlight Radiation)

Figure 3 shows the variation of sunlight radiation on  $\text{SnO}_2$  sample (growth temperature  $100^\circ\text{C}$ ) for four consecutive sunny days referred to as “peak sun hours” which is solar insolation at a particular location during the sun shining at its maximum level for a certain time. The distance from the surface of the sample parallel to the solar power meter was 65cm. The duration of sunlight radiation measurement for each sunny day was 15 minutes. Since the peak solar radiation is  $1 \text{ kW/m}^2$ , the percentage is the received solar radiation by the tin oxide sample over the peak solar radiation. The intensity of sunlight radiation at day one was  $125.0 \text{ W/m}^2$  with temperature  $36^\circ\text{C}$  which results in 12.5% of solar radiation. The intensity of sunlight radiation at day two was recorded  $189.9 \text{ W/m}^2$  which resulted 18.9% of solar radiation. The sunlight radiation was also affected by the weather of the day. Temperature at day two was recorded as highest which is  $38^\circ\text{C}$ . At day three, the temperature was recorded  $34^\circ\text{C}$  and the intensity of sunlight radiation reading showed lower value  $69.2 \text{ W/m}^2$  which results in 6.9% of solar radiation. The lowest intensity reading was recorded at day four with radiation

55.5 W/m<sup>2</sup> and the solar radiation percentage of 5.5%. This is due to the fact that at day four, the lowest temperature was recorded 32°C.



**Figure 3.** Variation of sunlight radiation on SnO<sub>2</sub> for four sunny days (growth temperature 100°C).

#### 4. Conclusions

SnO<sub>2</sub> nanostructures were successfully prepared using thermal oxidation method at growth temperature 100°C and growth time of 1 hour in tube furnace. The FESEM analyses showed that the average particle size diameter of SnO<sub>2</sub> varies from 97.5nm to 142nm compared to Sn particle size which varies in the range of 5.88µm to 7.43µm. The agglomeration was occurred during the thermal oxidation of the SnO<sub>2</sub> at 100°C oxidation temperature. The sunlight radiation readings on SnO<sub>2</sub> sample (growth temperature 100°C) were measured for four consecutive sunny days and the radiation 189.9 W/m<sup>2</sup> was recorded at day two for the daytime temperature 38°C. At day two, the highest solar radiation percentage was measured 18.9%.

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