THE EFFECT OF THE POST-ANNEALING TEMPERATURE ON THE NANOSTRUCTURE OF SNO₂ SYNTHESIZED BY THERMAL EVAPORATION METHOD
L.S. Chuah

Physics Section, School of Distance Education, Universiti Sains Malaysia, 11800 Penang, Malaysia.

ABSTRACT
In this study, tin dioxide (SnO₂) thin films were deposited on the silicon substrate using thermal evaporation method. This study proposes a low-cost thermal evaporation method to fabricate different forms of SnO₂ structures and it is expressly beneficial for large area of nanotechnology utilizations. This novel process is fast and can be completed at lower temperature. In addition, thermal evaporation deposition has the advantage over the other methods in that it does not take much time and is a template-free method to prepare SnO₂ nanostructures. After the tin (Sn) powders evaporate, the silicon substrate was cooled down and then removed to furnace (under air flow). All sample substrates were annealed in air with difference temperatures (400 °C, 500 °C, 600 °C, 800 °C, 1000 °C) in furnace. The degree of crystalline strongly increases by increasing the annealing temperatures. All sample layers were translucent in visible range and so do the white layer on the substrate. The morphology the SnO₂ thin films were studied using X-ray diffraction (XRD) and scanning electron microscopy (SEM).

Keywords -- Thermal Annealing; Particles; Tin Powder

I. INTRODUCTION
Tin oxide (SnO₂) is an oxygen defect n-type transparent conducting material, which shows high infrared reflectance in addition to high visible light transmittance (80-90%) because of the wide band gap. It has been extensively applied as heat mirrors, transparent electrodes for solar collectors, optoelectronic devices, heat mirrors, gas sensors, catalyst supports, and solar cells [1-4]. Up-to-date, attention has concentrated on the utilizations of a SnO₂ transparent electrode to electronic devices for example electrochromic, electroluminescent and gas sensing [5,6].

Nowadays, different methods can be applied to prepare tin oxide films on several different substrates. The synthetic methods for SnO2 nanostructures include laser ablation, reactive RF sputtering, dip deposition, carbothermal reduction, chemical vapour deposition (CVD), spray pyrolysis, electro deposition, solvothermal synthesis, thermal oxidation, sol-gel dip coating, dc magnetron and thermal evaporation [7-9]. Among these, it is well known that thermal evaporation method does not require sophisticated instruments such as high vacuum systems. The thermal evaporation is the most popular route for nanostructures because of it simplicity, low cost and readily available and the parameters are easily controlled. SnO films can be deposited on different substrate materials such as glass, stainless steel, aluminium, silicon, Al₂O₃ and niobium [10-12].

There are some literature reported the preparation of SnO films using thermal evaporation methods. According to Cakmak et al., they studied the consequence of thermal oxidation temperature on tin dioxide,
SnO$_2$ films were synthesized on quartz substrates by vacuum evaporation of tin metal. The results revealed that the films are included of nanostructured particles, show good crystallinity. Growth parameters for examples temperature, carrier gas flow rate, and substrate play a important role in determine the surface morphology nature and characteristics of the nanostructures. Optical band gap of the SnO$_2$ films was raised with increasing annealing temperature. In present work, we study the evaporation method affect the crystalline properties of the tin oxide films in terms of crystallinity and crystalline size.

II. EXPERIMENT

Silicon (Si-111) substrate was applied for the growth of the SnO$_2$ nanostructures. First, the substrate was cleaned in RCA. The thermal evaporation technique was used to deposit the Sn (99.9 %) thin films with thicknesses 2500 Å on silicon substrate. All sample substrates were annealed in air with difference temperatures (400 °C, 500 °C, 600 °C, 800 °C, and 1000 °C). Next, the sample is loaded in high temperature furnace and annealed at atmospheric pressure. Finally, the samples are cool down to room temperature and then unloaded. The morphological changes of as-synthesized SnO$_2$ thin films were observed via a scanning electron microscope (SEM) (JSM 6460LV, Jeol Inc.), Next, the samples were characterized by using energy dispersive X-ray spectroscopy (EDX), and X-ray diffraction microscopy (XRD) for extracting their elemental composition and crystallographic nature.

III. RESULT AND DISCUSSION

Figure 1 shows a typical EDX spectra of uncoated Si samples annealed at 1000 °C for two hours in the air ambience. Energy dispersive X-ray (EDX) analysis (in the SEM) was used to test the composition of SnO$_2$ microstructures. Figure 1 indicates the presence of Sn and O in the nanostructures. No other metal elements are aiding in catalyzing the observed structures growth. The peak intensity corresponds to the concentration of a particular element in the sample (the higher the concentration, the higher the peak). Apart from that, the peak intensity also depends on the weight of the element (the heavier the element, the higher the peak) and kinetic energy (high energy has a larger penetration depth).

![Fig. 1. Typical EDX spectrum of the as synthesis SnO$_2$.](image)

The surface morphologies of the SnO$_2$ films changed with an increase in oxidation durations, as shown in Figure 2. The SEM image looked smooth and composed of smooth surface area with well defined grain boundaries over the substrate surface without any pinholes and cracks. As the increasing annealing temperature, well-developed micro-crystallites phases. The thermal annealed route would influence the amorphous compounds, crystal growth and the agglomeration of similarly precipitated tin powders. Therefore, the thermal annealing
method could be proposed as a unique route to get an absolutely high surface area of crystalline tin oxide powders for apply in different utilizations [13]
Fig. 2. SEM surface morphology of SnO thin films annealed at difference temperatures for various durations.

The XRD patterns of prepared SnO\textsubscript{2} nano-powders at various annealing temperatures are revealed in Figure 3(a)-3(c). It is obvious that the samples annealed at 400, 500, 800, 1000 °C are completely crystallized. The XRD patterns at all annealing temperatures show that the intensities of three fundamental peaks corresponding to the (110), (101) and (211) planes are more than of other peaks.

The mean size of nanocrystallite was also estimated using the Scherrer equation based on the XRD patterns [1],

\[ D = \frac{K\lambda}{\delta w \cos \theta}, \]  

where D is the mean size of particle, K is a constant (around 1), \( \lambda \) is the X-ray wavelength, \( \delta w \) is the full width at half maximum (FWHM) of XRD peaks and \( \theta \) is the Bragg’s angle. The temperature dependence of particles size in crystalline orientations of (101). The mean size of nanoparticles has increased about 50 - 70 nm with increase in annealed temperature.
Fig. 3. The XRD patterns of the SnO2 nano-particles annealed at different temperatures, (a) 400 °C, (b) 500 °C, and (c) 1000°C.

IV. CONCLUSION
Nanostructured tin oxide films have been deposited on glass substrates at room temperature using thermal evaporation technique and following thermal oxidation was performed under constant air flow at various temperature. The results revealed that the films are included of nano-sized particles, show good crystallinity and work as a semiconductor material. This study exhibited that evaporated SnO thin films which were coming after thermally oxidized could raise the efficiency of SnO films in current utilizations.
ACKNOWLEDGEMENT

Financial support from short term grant (304/PJJAUH/6311052) from Universiti Sains Malaysia are gratefully acknowledged.

REFERENCES: