



## Investigation of Single-Crystalline SnO<sub>2</sub> Bipyramidal: Synthesis and Properties

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### Abstract:

Semiconductor nanostructure metal oxides have attracted a lot of attention due to their unique electrical, optical, and mechanical properties and potential application over their bulk counter parts. Among them, Tin Oxide (SnO<sub>2</sub>) nanostructures, as an n-type semiconductor with a direct wide band gap ( $E_g = 3.6$  eV at 300K,) and high exciton binding energy (130 meV) have been intensively investigated because of anticipated application in many areas. However, it should be noted that the size and morphology of the material greatly affect their properties and applications. SnO, as an important functional material with various applications, such as gas sensors, solar cells, transparent conducting electrodes and in catalysis, were synthesized using simple electrolysis of metal electrode in water at different voltages with ionic conductivity-108 x 10<sup>3</sup> Siemens and pH 7.85 as electrolyte. The crystal structure, surface morphology and chemical composition of the as synthesized product were analyzed using XRD, SEM and EDAX techniques respectively. In the present study, we report the synthesis and characterizations of nanostructure SnO, bipyramids.

**Keywords:** Nanostructured, Bipyramidal, Semiconductor Oxides, Tin Oxide

## 1. Introduction

One-dimensional (1D) nanostructured metal oxides have gained significant attention in recent years due to their enhanced physical and chemical properties compared to their bulk counterparts. Among these materials, tin oxide (SnO<sub>2</sub>) is considered one of the most important transparent conducting metal oxides [1]. In its nanostructured form, SnO<sub>2</sub> exhibits remarkable electrical, optical, and sensing properties, making it highly suitable for a wide range of applications such as solar cells, gas sensors, and transparent conducting electrodes [8–10]. The performance of these nanomaterials is strongly influenced by their size, shape, and morphology, which play a crucial role in determining their functional characteristics and practical applications. Recently, considerable research interest has been focused on tin oxide due to its excellent mechanical strength, chemical stability, environmental compatibility, and good thermal properties [4]. In its bulk form, SnO<sub>2</sub> possesses a tetragonal rutile crystal structure with lattice parameters  $a = b = 4.737$  Å and  $c = 3.186$  Å. It has a wide direct band gap of approximately 3.67 eV at room temperature (300 K) [2] and a high exciton binding energy [3], which contribute to its semiconducting behavior. Although it behaves as an insulator under certain conditions, SnO<sub>2</sub> typically exhibits n-type semiconducting properties due to oxygen vacancies. SnO<sub>2</sub> is widely used as a transparent conducting oxide (TCO) material because of its excellent optoelectronic properties. Various synthesis methods have been developed to fabricate SnO<sub>2</sub> nanostructures with different morphologies, including nanoparticles,

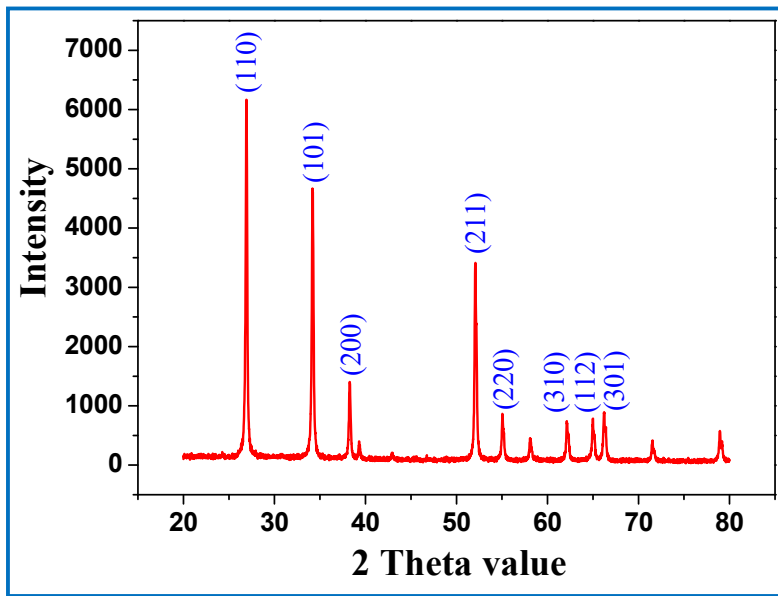
nanowires, nanorods, and bipyramidal structures. Among these techniques, the hydrolysis method is considered one of the most promising approaches due to its simplicity, low cost, environmentally friendly nature, and suitability for large-scale production [6]. In the present work, we report the large-scale synthesis of single-crystalline SnO<sub>2</sub> nanobipyramids using a hydrolysis route. The synthesis process utilizes water and ethanol as environmentally benign electrolyte and washing solvents, respectively. This method provides an efficient pathway for producing well-defined nanostructures with controlled morphology, which can significantly enhance their potential applications in advanced electronic and sensing devices.

## **2. Materials and Methods**

The synthesis of SnO<sub>2</sub> nanostructures was carried out using a simple two-electrode electrochemical setup with 10 ml of plain distilled water as the electrolyte. A high-purity tin sheet (Sigma Aldrich, 99.99%) of size 2 × 1 cm was used as the sacrificial anode, while a platinum sheet of the same dimensions served as the cathode. Prior to electrolysis, the tin electrode was polished to obtain a mirror-like surface and cleaned ultrasonically in ethanol followed by distilled water to remove any oxide layer and impurities. The electrodes were placed at a fixed distance of 2 cm in a glass electrolysis cell containing distilled water with low conductivity ( $\sim 1.08 \times 10^{-3}$  S) and pH 7.85. Electrolysis was performed at different applied voltages (2–10 V) for varying durations. The resulting white precipitate was collected by centrifugation, washed thoroughly, and dried at 60°C for 5 hours.

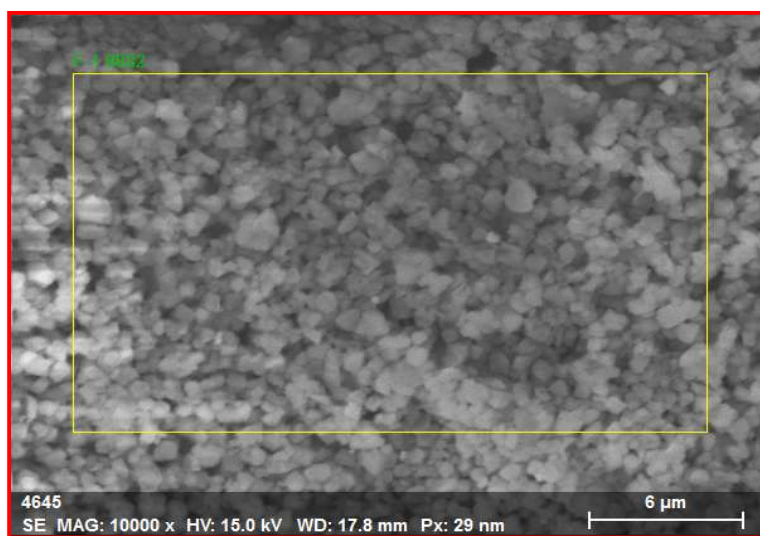
## **3. Results**

The structure of as obtained product was characterized by X-ray diffraction (XRD). The X-ray diffraction pattern was recorded on a powder sample with diffractometer (Model-D8 Advance, Bruker AXS) employing Cu K $\alpha$  radiation ( $\lambda = 1.540 \text{ \AA}$ ) as a source at a  $2\theta$  ranging from 20 to 80°. The diffraction pattern in figure 1 of as obtained product at 10 volt shows orientation in (110) and (101) direction and all other peaks can be well indexed to tetragonal SnO<sub>2</sub> with rutile structure. The surface morphology of the as obtained product at different applied voltages has been investigated using Scanning Electron Microscopy (SEM) (JEOL, JSM-6360 A). Figure 2 is a SEM image of as obtained product after hydrolysis at 10V for 6 hours and product collected from bottom of the cell. As seen from SEM image these structures have morphology like bipyramids of uniform size.



**Figure 1:- X-ray Diffraction pattern of SnO<sub>2</sub>**

The as-prepared SnO<sub>2</sub> was prone to growing into a square pyramid, its thermodynamically stable morphology.



**Figure 2:- Scanning Image Microscopy of SnO<sub>2</sub>**

Similar structures were also obtained for 2 V and 4V. The chemical composition of as obtained product was analyzed by energy dispersive X-ray analysis (EDAX), figure 3 show the composition pattern of SnO<sub>2</sub>. It indicating the presence of tin (Sn) and oxygen (O) as the primary constituents. The quantitative analysis reveals that oxygen contributes approximately 92.89 wt.% while tin accounts for about 7.11 wt.%, suggesting the formation of SnO<sub>2</sub>. The prominent peaks corresponding to O (K-series) and Sn (L-series) validate the successful synthesis of tin oxide material. No significant impurity peaks are observed, indicating high purity of the sample. The slight deviation in atomic percentages may be attributed to instrumental limitations or surface effects.

Overall, the EDS results confirm the stoichiometric formation and compositional uniformity of the prepared SnO<sub>2</sub> nanostructured material.

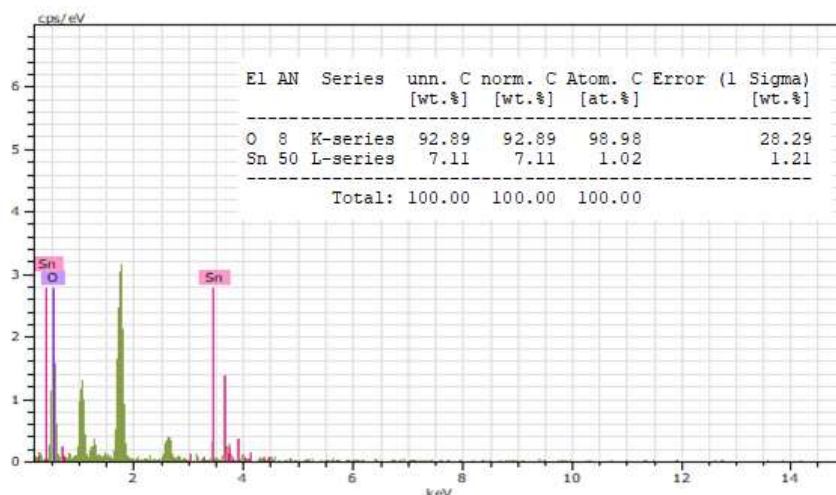


Figure 2:- Elemental Dissipative Analysis of SnO<sub>2</sub>

#### 4. Conclusions

In this study, single-crystalline SnO<sub>2</sub> bipyramidal nanostructures were successfully synthesized using a simple and eco-friendly electrolysis (hydrolysis) method. The technique employed plain water as an electrolyte, making the process cost-effective and environmentally benign. XRD analysis confirmed the formation of tetragonal rutile SnO<sub>2</sub> with preferred orientations along the (110) and (101) planes, indicating good crystallinity. SEM analysis revealed well-defined bipyramidal morphology with uniform size distribution, demonstrating controlled growth of nanostructures. EDAX results verified the presence of only tin and oxygen elements, confirming the purity and stoichiometric composition of SnO<sub>2</sub>. The synthesized nanostructures exhibit promising structural and morphological characteristics suitable for applications in gas sensing, optoelectronic devices, and catalysis. Overall, the study highlights an efficient route for large-scale production of SnO<sub>2</sub> nanostructures with controlled morphology, which can significantly influence their functional properties and enhance their applicability in advanced technological applications.

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