



## **Effect of annealing temperature on physical properties of tin oxide nanoparticles by microwave assisted route**

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**Abstract** - In the present work, nanostructured SnO<sub>2</sub> nanoparticles were synthesized by microwave assisted technique and the synthesized samples were annealed at two different temperatures (600°C & 700°C) for analysis. The X-ray diffraction pattern revealed the tetragonal rutile phase of SnO<sub>2</sub> and average crystalline size was found to be in the range of 26-29 nm. Functional groups were analyzed by FTIR. Raman vibrational modes attained the rutile phase of SnO<sub>2</sub>. The Photoluminescence spectra is used to confirm the presence of structural defects such as oxygen vacancies and metal interstitials. EDS spectra confirmed the presence of Sn, O in SnO<sub>2</sub> nanoparticles

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**Keywords:** SnO<sub>2</sub> microwave, tetragonal, bluish green emission.

### **I. INTRODUCTION**

In recent years, the synthesis and physical characterization of nanostructured semiconductors have found much interest. Among these semiconductors, SnO<sub>2</sub> has attracted great attention due to its optical and electronic properties [1]. Rutile SnO<sub>2</sub> is an n-type semiconducting oxide with wide range of energy gap ( $E_g = 3.62\text{eV}$ ), which has been used in large range of applications such as gas sensor, transparent conducting electrodes, rechargeable Li batteries and optical electronic devices [2]. Many methods have been used to develop SnO<sub>2</sub> nanostructures such as sol-gel, precipitation, hydrothermal and solvothermal, etc. Among these, microwave assisted synthesis technique has acquire lot of advantages to produce nanomaterials with uniform structure in large scale. In the present work, we have synthesized SnO<sub>2</sub> nanoparticles and studied its physical properties on influence of annealing temperature.

### **II. EXPERIMENTAL PROCEDURE**

Initially, 0.1M SnCl<sub>2</sub>·2H<sub>2</sub>O was dissolved in 100 ml distilled water and the calculated amount of ammonium hydroxide was added dropwise until it reaches to pH 8. After completion of the reaction white precipitation was obtained. The white precipitation was collected by centrifugation and washed with water and ethanol several times. The final product was transferred into polypropylene capped autoclave bottle. The autoclave bottle was kept in a microwave oven and treated at 600W for 5 mins. The final product was dried at 100°C in hot air oven for 12 hours and annealed at 600 and 700°C for 5 hrs.

### **III. RESULTS AND DISCUSSION**

#### **X-Ray Diffraction:**

X-Ray Diffraction is one of the most important tool to characterize the crystalline structure of the materials. Figure 1 shows the XRD pattern of SnO<sub>2</sub> nanoparticles annealed at different temperatures, which reveals the tetragonal rutile phase of tin oxide with space group P42/mmm. Further, the obtained peaks were indexed to (110), (101), (200), (111), (211), (220), (002), (202), (321) plane of SnO<sub>2</sub> which are in close agreement with the standard JCPDS file No.41-1445 [3]. The average grain size increases from 26 to 29 nm and the crystal defects such as dislocation density and micro strain values are found to decreased with the increase of annealing temperature from 600 to 700°C.

#### **FTIR Analysis:**

FTIR spectrum is used to analyze the functional group of synthesized nano materials. FTIR spectra of annealed SnO<sub>2</sub> nanoparticles are recorded and are shown in figure 2. It is observed that the absorption peak found at 3425 cm<sup>-1</sup> is attributed to the hydroxyl group vibration of SnO<sub>2</sub> nanoparticles due to the incorporation of water from ambient atmosphere[4]. The weak peak found at 2923 cm<sup>-1</sup> is attributed to stretching vibration C-H bonds. The peak at 1634 cm<sup>-1</sup> may be due to the absorption of water and ammonia[5]. The formation of SnO<sub>2</sub> nanocrystals is confirmed from the peak to at 623 cm<sup>-1</sup>.

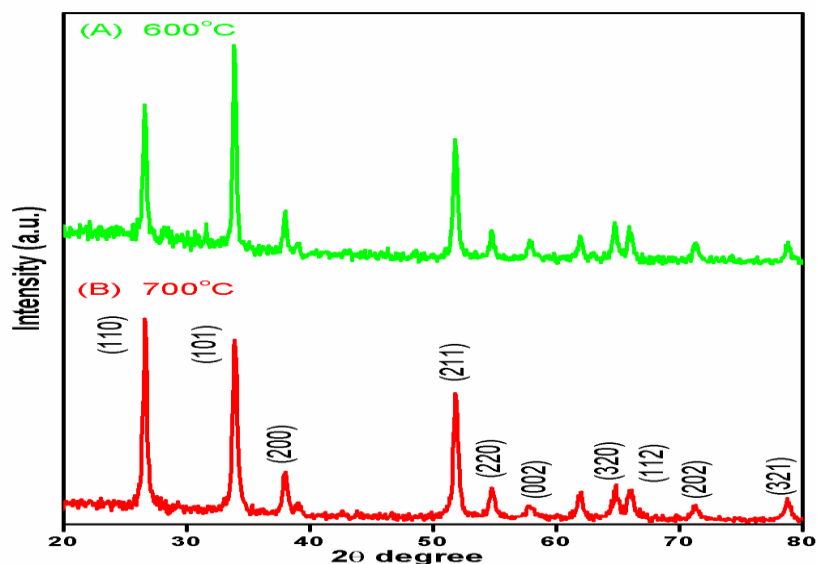


Figure 1. XRD pattern of SnO<sub>2</sub> nanoparticles annealed at different temperatures

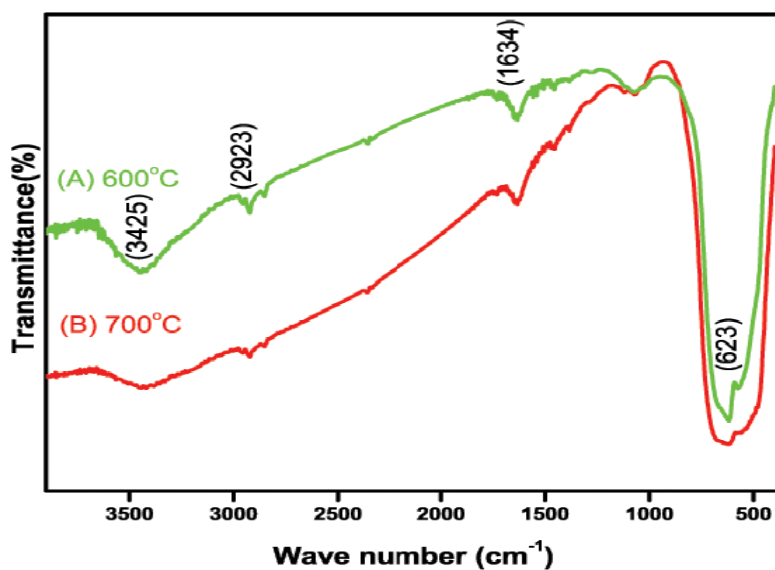


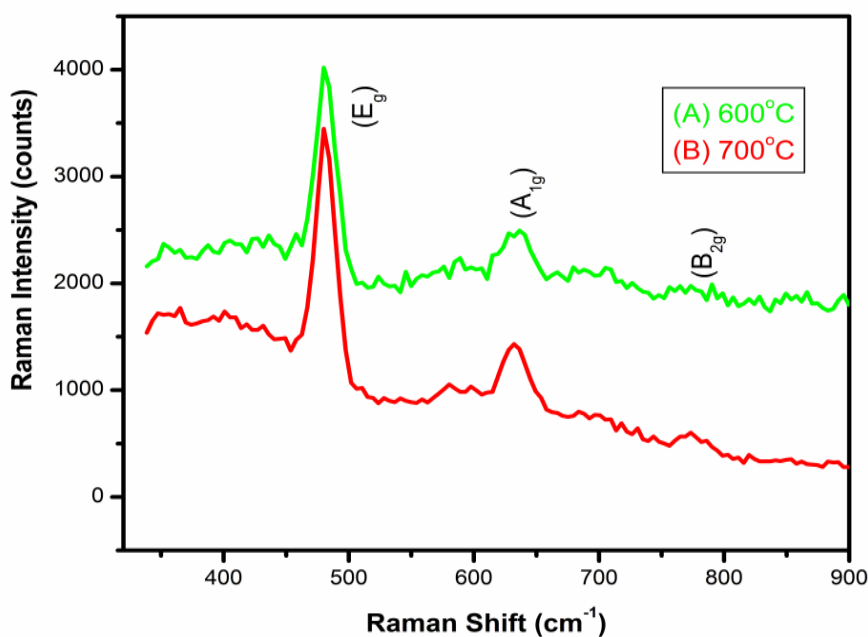
Figure 2. FTIR Spectra of SnO<sub>2</sub> nanoparticles annealed at different temperatures

#### Raman scattering analysis:

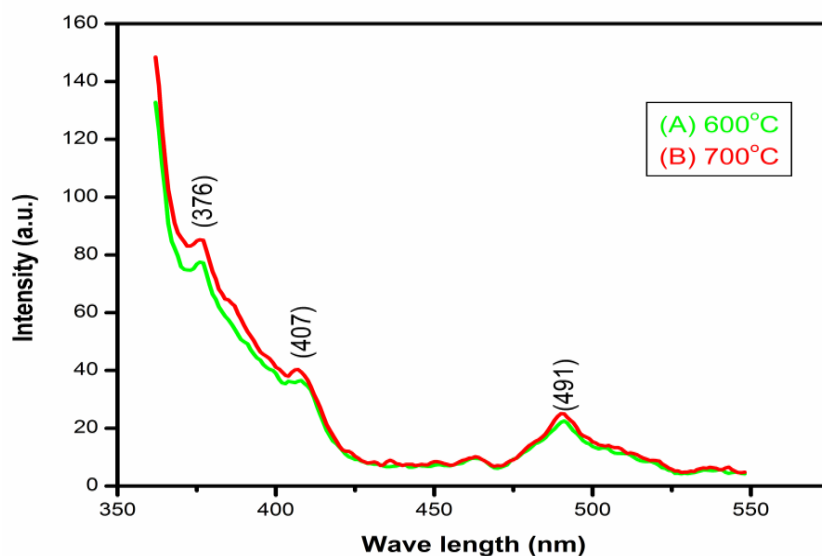
Raman scattering is used to analyze the vibrational modes and phase confirmation of synthesized nanoparticles. Figure 3 shows Raman spectra of SnO<sub>2</sub> nanoparticles. The vibrational Raman modes assigned at 633, 475 and 775 cm<sup>-1</sup> corresponds to A<sub>1g</sub>, E<sub>g</sub> and B<sub>2g</sub> modes of SnO<sub>2</sub> nanoparticles respectively [6]. These vibrational Raman modes confirmed the tetragonal rutile structure of SnO<sub>2</sub> and these are related to the vibration of oxygen in oxygen plane, expansion and contraction of Sn-O bonds [7].

#### Photoluminescence analysis:

Figure 4 shows the photoluminescence (PL) spectra of SnO<sub>2</sub> nanoparticles. PL emission bands are located at 376, 407 and 491 nm. Further, the intensity of luminescence spectra was varied with respect to the annealing temperature. Near band edge emission related peak of 376 nm is assigned in the UV region [8]. The emission peak at 407 nm is ascribed to the structural defects of tin interstitial or dangling in the material. The transition between the oxygen vacancy and photoexcited holes related to the bluish green emission peak is found at 491 nm [9].



*Figure 3. Raman spectra of SnO<sub>2</sub> nanoparticles annealed at different temperatures*



*Figure 4. Photoluminescence spectra of SnO<sub>2</sub> nanoparticles annealed at different temperatures*

#### **FESEM and EDX analyses:**

The surface morphologies of annealed SnO<sub>2</sub> nanoparticles were analyzed by using FESEM. and the images are shown in figure 5. It is found that an agglomerated spherical sized nano particles which cover the entire surface uniformly. The EDX spectra confirms the presence Sn and O in SnO<sub>2</sub> nanoparticles which confirms the high purity of SnO<sub>2</sub> nanoparticles.

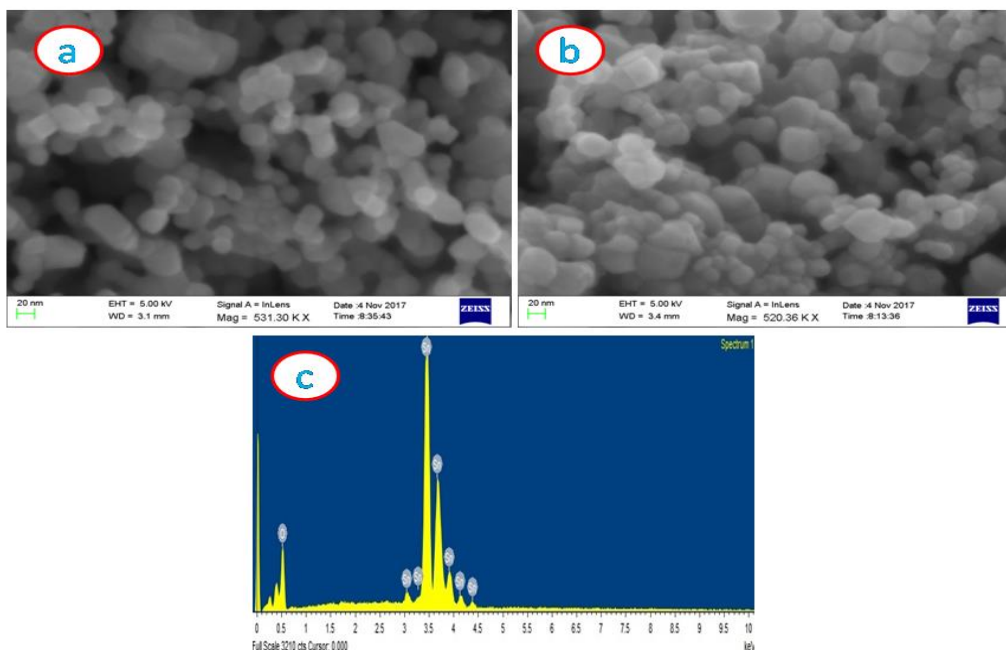


Figure 5. SEM and EDS spectrum of annealed SnO<sub>2</sub> nanoparticles

#### IV. CONCLUSION

In summary, SnO<sub>2</sub> nanoparticles have been successfully synthesized by the microwave assisted method. The effect of annealing temperature on structural and optical properties of prepared SnO<sub>2</sub> nanoparticles were studied. Increased crystallite size and ased dislocation density and micro strain were found with the increase of annealing temperatures. Different emission spectra such as UV, bluish green emissions were observed from the PL spectra. From these results one can conclude that SnO<sub>2</sub> nanoparticles annealed at 700°C is having the improved structural and optical properties.

#### ACKNOWLEDGMENTS

The author S.Asaitambi gratefully acknowledge for financial support for this work by DST-PURSE programme (Ref: Rc.S.O.(P&D)/DST-PURSE Phase II/10815/2017).

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