

## Growth and characterization of optical structural and morphological studies of ZTO through chemical method

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### Abstract

In current study for the first time, we were able to successfully synthesize single-crystalline ZTO nanorods using a straightforward microwave irradiation technique. ZTO nanorods have a length of up to several micrometers and a diameter of 25-50 nm and are single-crystalline, structure obtained from XRD. UV-VIS diffuse reflectance spectroscopy was used to examine the optical properties. The photoluminescence spectrum (PL) of the nanorods at room temperature shows stable broad blue-green emissions around the 400–600 nm wavelength range, with a maximum center at 490 nm and 520 nm. This is in good agreement with the reported values of 3.25 eV. ZTO nanorods have potential applications in high-performance supercapacitors, according to these findings. faradaic method of reaction

Key words: PL, UV, nono rod, nanowire, etc.,

### Introduction

Nano-scale means something that is one billionth of a meter i.e.  $1\text{nm} = 10^{-9}\text{m}$ . Any material termed as nano material if one of its dimensions is in nanoscale (preferably less than 100 nm). Nanomaterials driven technology is referred as nanotechnology; it connects different fields of sciences (physics, chemistry and materials sciences). Our Earth's crust

has abundance of metal oxides like  $\text{Fe}_3\text{O}_4$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{ZnO}$ ,  $\text{CaO}$  etc. even the pure elements get oxidized in the presence of air and water to form oxides. Chemical compounds containing one oxygen anion in the -2-oxidation state and other element in its formula are termed as Metal-Oxides. Oxides of metals has occupied significant space in many areas of material science, physics and chemistry due to its wide range of properties. Our daily life owe a lot to metal oxides due to its different properties from touch screens of our mobile to the aluminium foil for wrapping food almost every corner of our routine is bonded with metal oxides. Metal oxides exists in variety of crystal structures electronic and optical properties which gives them metallic, semiconducting or insulating characteristics accordingly. Fascinating properties like ferroelectricity, multiferroic, high temperature superconductivity are exhibited by metal oxides making them multifunctional. Our modern lifestyle seeks the development of materials that are having multiple applications and stimulates the research developments towards this direction.  $\text{SnO}_2$ ,  $\text{ZnO}$ ,  $\text{TiO}_2$  and  $\text{Fe}_2\text{O}_3$  are binary multifunctional metal oxides that are already being used as sensors, electrodes and varistors. Besides these binary oxides, we also have ternary and complex multifunctional metal oxides that serve the same purposes. As per requirement of the application these materials can be synthesised in different morphologies by keeping these in nanostructures, bulk or thin films [5]. In semiconductors, band gap is a crucial parameter for modifying optical and electrical properties and there exists several categorizations on the basis of type of band gaps (Direct or Indirect). These days, we are utilising the benefits of wide band gap semiconductors in our day-to-day life e.g. in touch screens, LCDs, sunscreens, in purifying water, gas sensing, Solar cells etc.

#### **Zinc stannate ( $\text{Zn}_2\text{SnO}_4$ )**

Zinc stannate ( $\text{Zn}_2\text{SnO}_4$ ) has attracted a lot of attention due to its potential uses in transparent conducting electrodes, photo catalysts, sensors, Li-ion batteries, and solar cells.  $\text{Zn}_2\text{SnO}_4$  has been one of the most active areas of research in the field of optoelectronic materials ever since it was first described as the photo anode of a dye-sensitized solar cell (DSSC). The electron mobility of  $\text{Zn}_2\text{SnO}_4$  is  $10\text{--}15 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ , which is greater than that of typical binary oxide (e.g., Band gap of  $\text{TiO}_2$ : Electron mobility:  $3.2 \text{ eV}$   $105 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ ) indicates superior property for reducing dye photo bleaching caused by the UV spectrum of the sun.  $\text{Zn}_2\text{SnO}_4$  exhibits superior

acid media stability to ZnO, which results in the formation of the Zn-dye<sup>+</sup> complex, which reduces photovoltaic performance and photocurrent. By simply altering the ratio of Zn to Sn, the ternary oxide can also adjust its work function, band gap energy, and electric resistivity. Despite the many inherent advantages that Zn<sub>2</sub>SnO<sub>4</sub> has, it has not been extensively studied as a photo electrode in DSSC, unlike its competitors TiO<sub>2</sub>, ZnO, and SnO<sub>2</sub>. The majority of studies on Zn<sub>2</sub>SnO<sub>4</sub> photo electrodes used nanoparticles with an octahedral or 8–60 nm diameter, but power conversion efficiency was still below 4.7%. Poor results have recently been reported on the Zn<sub>2</sub>SnO<sub>4</sub> nanowire.

### **Materials**

SnCl<sub>2</sub>.2H<sub>2</sub>O and ZnCl<sub>2</sub>, deionized water for all the analytical reagents chemicals obtained from M/s E-Merck company , India .

### **Methods - Microwave Synthesis**

The process of heating with the microwave differs slightly from that of traditional heating. To begin, the microwaves must be able to pass through the reaction vessel almost completely. Only fluoropolymers and a small number of engineering plastics, like polypropylene and PEEK (polyether-ether-ketone) with glass fibers, are available as vessel materials. The vessel's surface is not where the reaction mixture is heated; The temperature of the vessel wall is almost always lower than that of the reaction mixture. In fact, the vessel wall can be a good way for the reaction mixture to lose heat.

Second, there must be a component of the reaction mixture that absorbs the penetrating microwaves for microwave heating to occur. The reaction mixture will be penetrated by microwaves, and the energy will become heat if it is absorbed. The reaction mixture can be mixed by convection, just like with traditional heating, or it can be mixed mechanically (stirring) to evenly distribute the temperature and reactants throughout the reaction vessel.

### **Synthesis of Zn<sub>2</sub>SnO<sub>4</sub> nanorods**

Without further purification, all reagents were analytical reagent grade. SnCl<sub>2</sub>.2H<sub>2</sub>O and ZnCl<sub>2</sub> are used in a typical experiment. 2H<sub>2</sub>O was dissolved in as little deionized water as possible in a ratio of 1:2 molar. After that, the NH<sub>3</sub>.H<sub>2</sub>O

solution was added drop by drop while vigorously stirring the mixture until the pH reached 8. An azure precipitate was produced after the reaction was completed. More than ten times, this precipitate was washed with water until the silver nitrate test revealed no chlorine ions.  $\text{NH}_4^+$  ions were removed from the precipitate by further washing it with ethanol. The resulting precipitate was irradiated for ten minutes in a teflon-lined household microwave oven (2.45 GHz) using up to 900W. At 80°C, the white precipitate was finally dried.

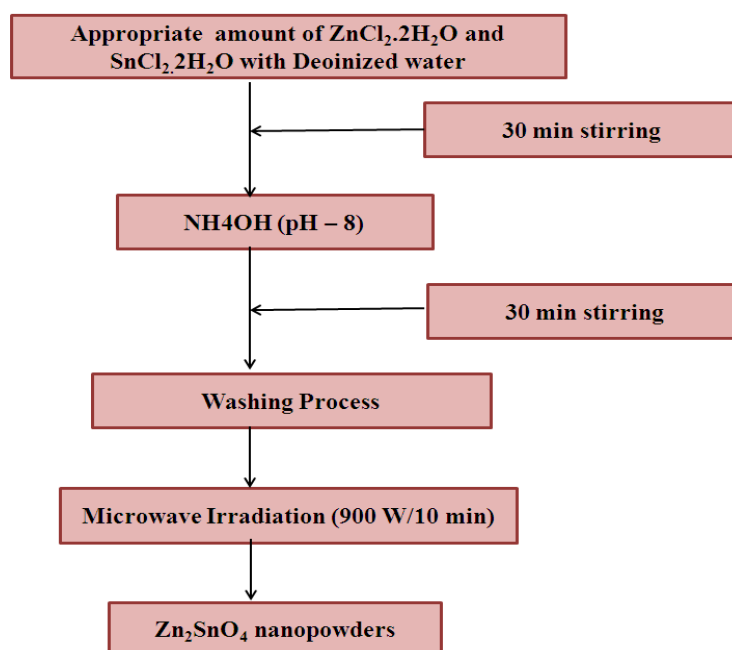


Figure 1- Schematic representation of experimental procedure

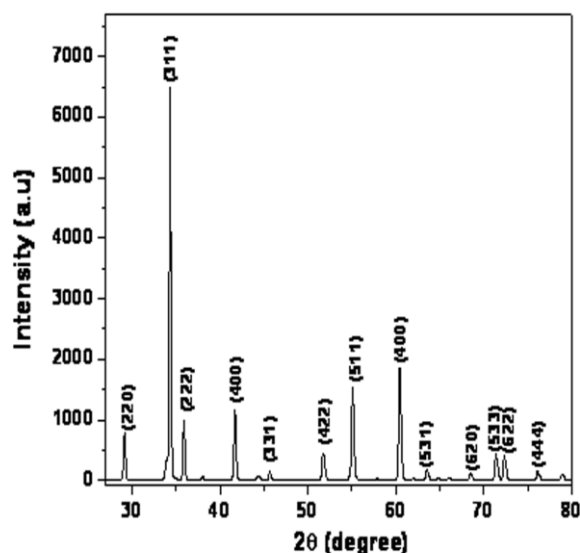
### Results:

**Structural studies obtained from XRD**, Transmission Electron Microscopy (TEM) and Selected-Area Electron Diffraction (SAED) patterns were recorded on a Technai G20-stwin Higher Resolution Electron Microscope (HRTEM) using an accelerating voltage of 200 kV. The optical properties were analyzed by UV-VIS diffusion reflectance spectroscopy using a CARY 5E UV-VIS-NIR spectrophotometer in the wavelength range of 200 – 800 nm.

### XRD analysis

XRD was used to determine the as-synthesized SnO<sub>2</sub> nanorods' structure and phase purity, as depicted in from the Fig.1. Face-centered spinel-structure ZTO can

be used to precisely index all of the sharp diffraction peaks. With calculated lattice parameters of  $a=8.6533$ , the miller indices [220], [311], [222], [400], [422], [511], [440], [531], [533], [622], and [444] are in good agreement.



**Figure 2 Powder XRD pattern of as-synthesized ZTO nanorods**

**TEM analysis** TEM micrographs were used to examine the nanorods' morphology as they were synthesized from the figure 3. The TEM image of ZTO nanorods at low magnification is shown in 2(a). and image showed a lot of nanorods. ZTO nanorods have a length of up to several micrometers and a diameter of 20-50 nm. Fig3.

#### HR-TEM

An HRTEM image taken from the nanorods' side edges is shown in Figure 5.2 (b). The (002) and (131) planes of the FCC ZTO structure are represented by two groups of parallel fringes with spacings of 0.43 and 0.26 nm, respectively. The high crystalline nature of the nanorods is confirmed by the HRTEM images' distinct lattice fringes. The direction in which the nanorods are growing can be indexed as [131]. Fig. also shows a corresponding SAED pattern of these nanorods recorded along the [310] zone axis. 5.2 (C). The crystalline ZTO nanorods have a growth direction of [133] as shown by the SAED pattern, which is in line with the HRTEM results.

### EDS analysis

EDS is used to examine the selected ZTO nanorods' composition. The atomic ratio of Zn/Sn/O 2:1:4 suggests that the sample is probably made of ZTO. Due to the grid used for EDS measurements, the composition contained the Cu element.

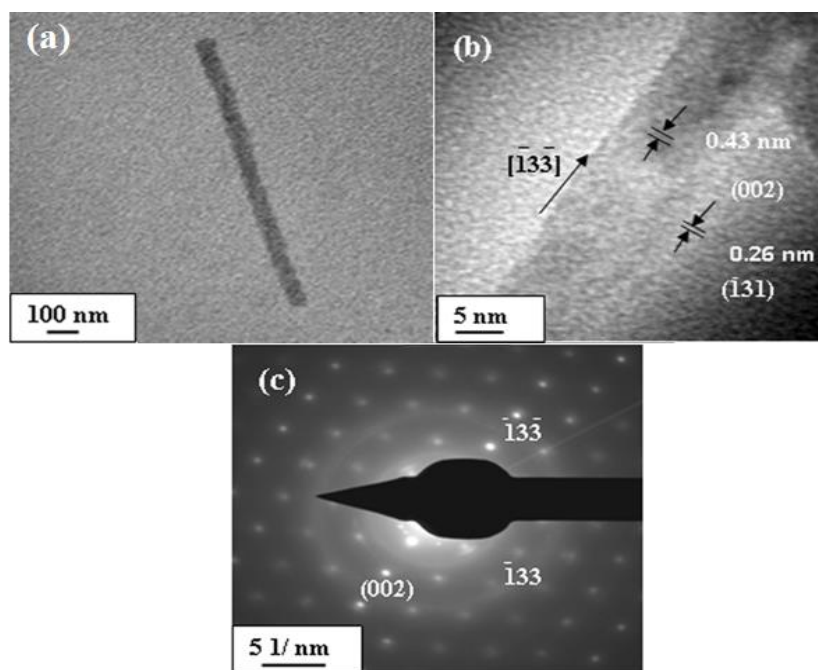


Figure 4. (a) low magnification TEM image of ZTO nanorods grows along [14]. (b) HRTEM image of ZTO nanorods. (c) Corresponding SAED pattern of ZTO nanorods, the SAED pattern was taken along the [310] zone axis.

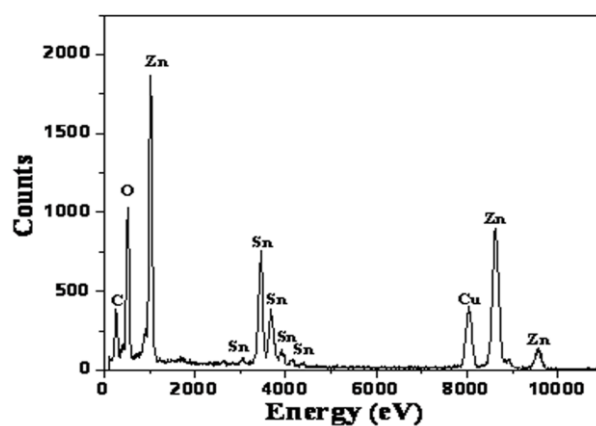


Figure 5 EDS spectrum of as-synthesized ZTO nanorods

## 6- UV-VIS diffuse reflectance spectroscopy

UV-VIS diffuse reflectance spectroscopy was used to examine the optical properties. Fig6. The diffuse UV-VIS reflectance spectra of ZTO nanorods as they are synthesized . The band gap energy serves as the intercept value on a graph that is drawn between  $[F(R)h]^2$  and  $h$  (fig.5). The as-synthesized ZTO nanorods have a band gap energy of 3.26 eV, which is consistent with the reported values of 3.25 eV [28].

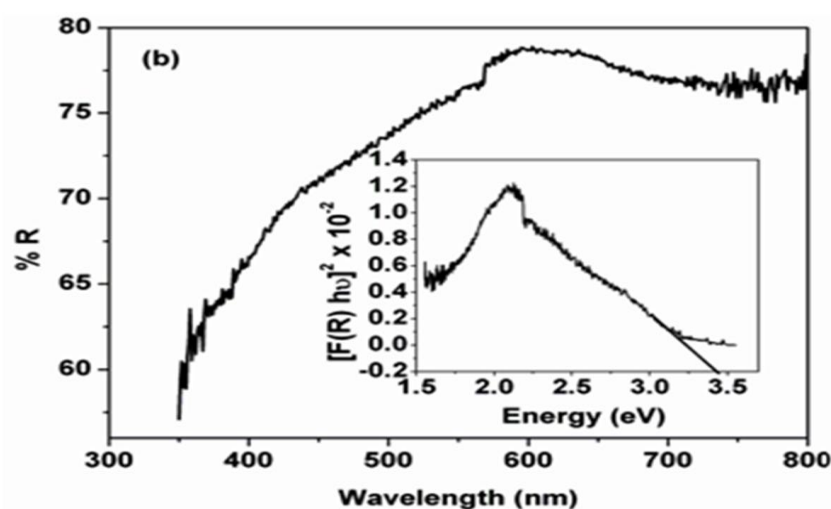


Figure 4. UV-VIS diffuse reflectance spectrum of as-synthesized ZTO nanorods

## 5 Photoluminescence spectroscopy

Photoluminescence (PL) analysis is an effective method for determining the crystalline quality of nanocrystals and the presence of defect structure. Fig 7 demonstrates the ZTO nanorods' photoluminescence spectrum as they are synthesized. At room temperature, the ZTO nanorods' PL spectrum was measured, and the excitation wavelength was 325 nm. Around the 400–600 nm wavelength range, the ZTO nanorods emit a stable broad blue-green spectrum with a maximum center at 490 nm and 520 nm. Evidently, the 360nm band-to-band emission peak is not the broad blue-green peak we observed. The PL mechanisms have always been attributed to other luminescence centers in previous studies of semiconductor 1D nanostructures, such as oxygen deficiency and residual strain during growth.

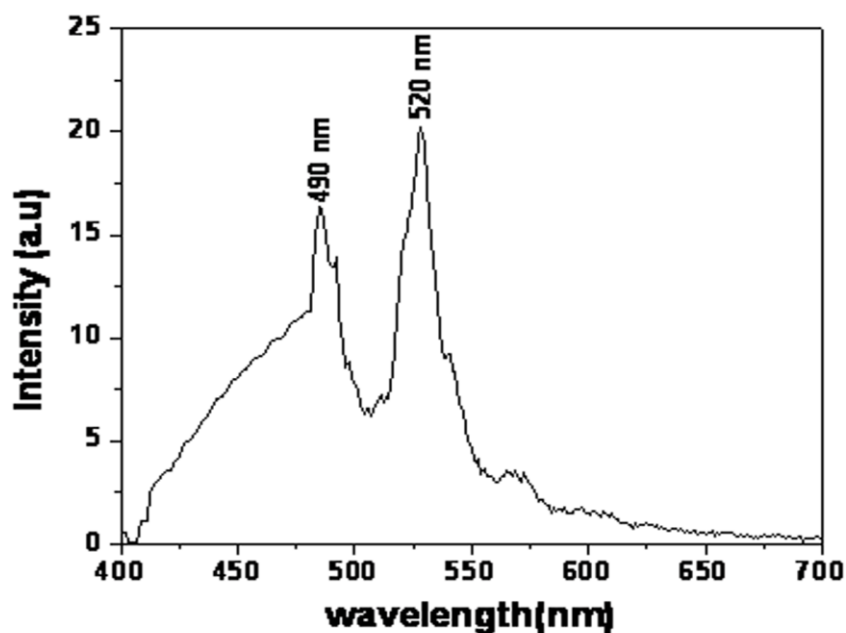


Figure 7

#### Photoluminescence spectrum of as-synthesized ZTO nanorods

The ZTO nanorods were prepared through microwave irradiation in our experiment. During the microwave irradiation process, oxygen deficiency will unavoidably generate oxygen vacancies. ZTO nanorods have the potential to be used in opto-electronic nanodevices and nanoscale smart devices, despite the fact that research into their properties is still ongoing.

#### CONCLUSIONS

In conclusion, for the first time, we were able to successfully synthesize single-crystalline ZTO nanorods using a straightforward microwave irradiation technique. ZTO nanorods have a length of up to several micrometers and a diameter of 25-50 nm. The ZTO nanorods have a growth direction of [133] and are single-crystalline, as shown by the selected area electron diffraction (SAED) pattern. UV-VIS diffuse reflectance spectroscopy was used to examine the optical properties. The photoluminescence spectrum (PL) of the nanorods at room temperature shows stable broad blue-green emissions around the 400–600 nm wavelength range, with a maximum center at 490 nm and 520 nm. This is in good agreement with the reported values of 3.25 eV. The nanorods' strong PL emissions have the potential to be used in nanoscale smart devices and opto-electronic nanodevices. At a scan rate of 10



mVs-1, the ZTO nanorods achieved a specific capacitance of 135 Fg<sup>-1</sup> and exhibit excellent electrochemical performance. ZTO nanorods have potential applications in high-performance supercapacitors, according to these findings. faradaic method of reaction.

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