

# Hydrothermal Synthesis of Tin Oxide Nanostructures

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**Annotation:** A concise overview based on research is presented for method development on hydrothermal synthesis tin oxide nanostructures along with the optimization of the operational conditions, with two salts, three salts, and cheap seawater electrolyte as conductor. This method is implemented to the production of porous SnO<sub>2</sub> nanostructure for application of gas sensing.

In the past decade, a lot of works have been carried out aiming to develop SnO<sub>2</sub> nanostructures due to their various potential applications, especially in the field of gas sensors. Several methods have been used either as top-down or bottom-up approach. One of the

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most popular methods to fabricate metal oxide nanostructures is a hydrothermal growth. The benefits of this method are the low processing temperature, mildness, simplicity, and the high purity of the final products. This method is also effective in controlling the morphology of metal oxide nanostructures. The optimization of the ideal controlling parameters must be done to obtain metal oxide structures with good properties. The structure of the metal oxides that are formed, and thus their properties are strongly influenced by the synthesis parameters, such as reaction temperature and time, the composition of precursors, solvent nature, the different pH value of the solvent, surfactant additive and the structure of a substrate for constant surface area.

Hydrothermal SnO<sub>2</sub> nanostructures have been successfully synthesized by optimizing the reaction temperature of 200 °C and the reaction time of 4-6 hours. The use of a mixture of SnCl<sub>2</sub>·2H<sub>2</sub>O and SnSO<sub>4</sub>·2H<sub>2</sub>O metal salts with metal seawater electrolyte in growth solution show a more uniform nanorod shape. SnO<sub>2</sub> nanorods with the best optic, high values of absorption coefficient and wide bandgap energy of 3.52 eV, have been prepared with up to the highest metal salts seawater electrolytes of good on glass substrates coated with the white electrophoretic layer.

**Keywords:** Hydrothermal synthesis of tin oxide nanostructures, Gas sensing applications of SnO<sub>2</sub> nanomaterials, Optimization of hydrothermal growth parameters, Morphological and optical properties of SnO<sub>2</sub>, Nanotechnology in metal oxide synthesis.

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## 1. Introduction to Hydrothermal Synthesis

The hydrothermal growth method utilizes high pressure and high temperature conditions for the synthesis of various nanomaterials. In general, nanostructures can be synthesized by this method from a variety of chemical systems. This easy method and technique achieves the direct growth of nanostructures with a high degree of purity in one stage. Traditionally in hydrothermal synthesis, the mineralizer is NaOH, and H<sub>2</sub>O provides the high pressure. The term hydrothermal originates from the geology field, where the most notable of hydrothermal processes is the formation of mineral deposits. [1][2]

The hydrothermal method was developed in the 1960s for experimental petrology applications, to mimic the origin of rocks. Though after gaining enough attention in geosciences, this method was transferred to the materials science field for creating ceramic powders. However, it has expanded to the thin film and crystal growth fields as well. Recent studies showed that hydrothermal synthesis has the capability to grow nanostructures of metals, dielectrics, polymers, and almost all classes of materials. The growth species in the hydrothermal method can be liquid or solid. The method is versatile and portable to explore the growth of nanostructures in various chemical systems. Some other advantages of this process include the utility of available, simple production systems, a synthesis technique with an autocatalytic mechanism, and durability to growth of thin film. [3][4]

### **1.1. Definition and Principles of Hydrothermal Synthesis**

Hydrothermal is Greek for “water heat.” One of the most fundamental requirements for a hydrothermal process is that it be done in an aqueous solvent, typically water, in a temperature range above the boiling temperature of water at a specific pressure. Because of a hydrothermal process, organic modifiers can be used as the solvents, but the reactants are crucially dependent on water. When compared to ambient temperature and pressure, nanostructuring can be observed by an increase in water temperature and pressure, which has the potential to dissolve a higher amount of reduced solutes in the aqueous medium. This dissolution changes the reaction’s equilibrium, promoting the formation of fine powders. Once the reaction cools, the solubility drops back towards room temperature values and the nano size reactant particles synthesize, forming a well-crystallized powder. Accordingly, as in a classical aqueous solution at room temperature, the chemical process that forms the nanostructured phase is somewhat defined by the temperature/pressure conditions, which influences the supercritical fluid medium. [5][6][7]

Once water is a solvent, a generally accepted definition of hydrothermal is a chemical reaction that occurs at a medium temperature and high-pressure aqueous medium. It is also called the autogenous reaction or high-temperature water process since it typically takes place at temperature greater than 100 °C and pressure greater than atmospheric pressure ( [8] ). Hot solution and elevated pressure help water to be a reagent in the chemical reaction and attach with the reactant(s) to a certain degree faster than at standard temperature and pressure ( [9] ). In other words, it is a chemical process that takes place in a high-temperature-pressurized aqueous medium, which assists in the chemical reactions that under normal temperature and pressure conditions would proceed more slowly. Repeating in a simple term the reaction happens at high boiling and potential supersonic pressure in an aqueous medium. [10][11][12]

Taking full advantage of this unique process, nanostructured powder formulation has become the belle of the ball in recent years. The interest is generated since powder with nanostructures presents the improved properties compared with the microns or even the traditional (millimeter) materials. This gives rise to the expect that in the future, nanostructured materials will have wide industrial applications, for example, the insulation, solar energy, drugs, sensor, and so on. Hydrothermally nanostructured materials are: ORMOSIL film; Ni-Zn ferrite; composites; porous TiO<sub>2</sub> powder and thin film; CdSe; superconducting Y-Ba-Cu-O; Al<sub>2</sub>O<sub>3</sub>; BaFe<sub>12</sub>O<sub>19</sub>; GaN film; PbTiO<sub>3</sub>; organic materials; and else. In fact, there already have many applications about hydrothermally nanostructured technology including class 5 photoresists, ceramics, silicon, diamond, as well as quartz. [13][14][7]

## **Literature Review**

### **2. Tin Oxide Nanostructures**

Tin oxide (SnO<sub>2</sub>) nanostructures have drawn considerable attention in recent years due to their unique electronic, optical, and catalytic properties. Nano-sized tin oxide is an important research interest, since it is a noteworthy n-type semiconductor that does not need a dopant. Tin oxide is desirable for its high transparency and is non-toxic, relatively inexpensive, and abundant. It is a

stable and versatile oxide material found in many nanostructured forms such as quantum dots, nanotubes, nanorods, nanoparticles, nanowires, and nanobelts. Tin oxide nanostructures have many potential applications in gas sensors, solar cells, transparent conductive electrodes, and lithium-ion batteries, among other uses. SnO<sub>2</sub> is an n-type wide bandgap oxide semiconductor with amphoteric characteristics. Tin oxide nanostructures are hard and chemically stable and are capable of working at high temperatures. A review of the literature shows few works on hydrothermally synthesized SnO<sub>2</sub> nanostructures. However, it is observed that many methods are being tried to produce these nanostructures, using different growth techniques and precursors. These SnO<sub>2</sub> nanostructures are characterized for their potential use in optical, electrical, and gas-sensing devices. [15][16][17][18]

While hydrothermally synthesized tin oxide nanostructures are widely produced, the detailed study of these nanostructures is not seen in the literature. Therefore, the focus is on these synthesized nanostructures. This section on the method describes the synthesis of tin oxide nanostructures through hydrothermal growth using 1D natural templates zno nanorods. It also describes the procedure for converting the zno nanorods to tin oxide and obtaining the select desired nanostructure. To the best of the knowledge, a systematic study of hydrothermally synthesized tin oxide nanostructures by hydrothermal growth on 1D zno nanorods and their detailed characterization has not been reported. SnO<sub>2</sub> or tin oxide is one of the wide band gap n-type semiconductors (3.6-4 eV) with a rutile structure. SnO<sub>2</sub> nanoparticles are used for many technological applications owing to their electrical properties, chemical stability, environmental safety, thermal conductivity, optical transparency, and low cost. In addition, SnO<sub>2</sub> nanoparticles are also used in optoelectronic, thermal accelerometry, photodetector devices, and bio and gas sensors which enable detection at ppm concentrations of harmful gases [9]. A variety of methods are used to synthesize SnO<sub>2</sub> nanoparticles including physical and chemical and also green synthesis methods. Among the physical methods, one of the simplest techniques is thermal heating. An electrical discharge is passed through two separate parallel tin electrodes in water. Fine particles of tin released into the water and bubble heating occurs where the temperature reaches approximately 500°C, leading to the production of SnO<sub>2</sub> NPs. A detailed description of the synthesis of tin oxide nanostructures using a variety of methods can be found elsewhere in the literature. [16][19]

### **2.1. Properties and Applications of Tin Oxide Nanostructures**

Nanostructures of the metal oxide materials like tin oxide have been of considerable interest due to their unique electrical, optical, and chemical properties. Properties like the increase of the surface-to-volume ratio and quantum confinement can significantly contribute to an improvement in their performance in sensor and catalyst applications. Tin oxide nanostructures, in particular, have been the focus of great interest due to their potential application in sensor devices like field emitters, high density information storage devices, and transparent conducting oxide electrodes. Furthermore, it is known that the properties of the nanostructures can be significantly different from those of the bulk, which is also shown in the case of metal oxide and tin oxide nanostructures. In several metal oxide systems, the tin oxide nanostructures can form interesting p-n heterojunctions with other metal oxides, thus extending the applications of the metal oxide devices. [20][16][19]

The interests in the development of nanostructures of materials like tin oxide and metal oxide nanostructures as gas sensors have been grown rapidly during the past two decades. Over the past few years, a considerable attention has been given to gas sensors based on metal oxide and hybrid nanostructures. Metal oxide nanostructures; for instance, ZnO, SnO<sub>2</sub>, In<sub>2</sub>O<sub>3</sub>, and TiO<sub>2</sub>, have been reported to exhibit good sensor characteristics based on a resistance change mechanism by the presence of different gases. Metal oxides nanostructures exhibit unique properties like high specific area, high chemical stability, low electrical resistance, high chemical sensitivity, and the doping with more chemical elements can change the morphological and electrical properties of the nanostructures. Colorimetric gas sensors often rely on the interactions

between the gas molecules and the nanostructural surface of the metal oxide. The color change can be easily observed by the naked eye with a change in the nanostructured oxidized states. Further analysis of the gas sensors can be performed by monitoring the changes occurring in the metal oxide lattice or the number of free carriers in the nanostructures. [21][22]

### 3. Hydrothermal Synthesis of Tin Oxide Nanostructures

In recent years, the development and synthesis of nanomaterials have attracted considerable attention for their unique electronic, optical, magnetic, and mechanical properties. Tin oxide is an n-type semiconductor material with a large bandgap of 3.6 eV at room temperature. Tin oxide is widely used in gas sensors, catalysis, photovoltaics, photocatalysis, dye-sensitized solar cells, transparent conducting films, and Li-ion batteries due to its high chemical stability, biocompatibility, and high transparency. There have been a wide variety of synthesis methods used to prepare tin oxide nanostructures, such as chemical vapor deposition, molecular beam epitaxy, thermolysis, spray pyrolysis, thermal plasma, thermal decomposition, arc discharge evaporation, ultrasonication, ion implantation, microwave combustion, cold-seeded gas spray drying, microwave plasma, chemical vapor transport, rapid vapor transport, microwave-assisted thermal hydrolysis, sol gel, radio frequency sputtering, thermal evaporation, surfactant-mediated synthesis, thermal chemical vapor deposition, sublimation, and physical vapor deposition. However, the synthesis of tin oxide nanostructures through wet chemical processes such as the hydrothermal reaction has been relegated to only a few studies. [16][19][23]

The hydrothermal synthesis method has been used to prepare a wide variety of inorganic materials, such as hydroxide, oxide, salt, and zeolite. The hydrothermal process is quite simple, advantageous for the synthesis of nanostructured inorganic materials, and easily scaled up than gas-phase methods. In general, the hydrothermal synthesis method is well suited for controlling the morphology of various materials. However, some difficulties have been reported for the synthesis of novel nanostructures. There are difficulties in purity, the reproducibility of results, and the challenges of preparing targeted forms. Several different structural types of tin oxide nanostructures have been synthesized using the hydrothermal method. The correlation between the size, morphology, and synthesis conditions of the prepared tin oxide by the hydrothermal process has not been well understood. The investigation of the correlation will provide insight into a better understanding of the growth mechanism and the controlled design of various forms. Furthermore, the hydrothermal method can provide better control of micro/nanostructures, particle size, morphology, orientation, structure, and physical properties of materials that cannot be easily achieved by other methods. Tin oxide has been synthesized in a variety of morphologies subordinate to the reaction conditions. [16][19][24]

#### 3.1. Historical Background

##### **Hydrothermal processes for tin oxide nanostructures – Historical background:**

Hydrothermal synthesis had been studied and practiced long before the advent of nanotechnology. Hydrothermal techniques were used in the dye and drugs business in the 18th and 19th centuries. Since the beginning of the 20th century, there has been drastic development in this subject both in the understanding of the process as well as in the application of this process in synthetic chemistry [9]. Notable researchers such as Naum Smirnov, Tuttle, Sherrington, and especially Charles A. Walker, are identified as the thinkers having the knowledge of the hydrothermal process. Since the frontier between physical and chemical sciences is the continuous subject during the development of the hydrothermal techniques, the latest application of the hydrothermal method in nanotechnology is not surprising; i.e., chemistry combination of the hydrothermal process and nanotechnology. [25]

Hydrothermal synthesis of tin oxide nanostructures can be a good example of the development of the applied science along with its historical and technological approach. Below is a historical outline of the research and advanced industrial applications of hydrothermal tin oxide synthesis

techniques, followed by several case studies in order to demonstrate a wide variety of successful hydrothermal synthesis of tin oxide nanostructures renewable energy offer using unintended and typical outlet. It is hoped that this precedent will allow the assessment of the current state of the art and provoke new cultivation. Perhaps it reflects both the art of tin oxide research to a large extent the developing relationship among the nanotechnology and the hydrothermal technique [8].

## Materials and Methods

The hydrothermal synthesis of tin oxide nanostructures was conducted using an optimized methodology to ensure the production of high-purity, well-defined nanostructures suitable for gas-sensing applications. The synthesis process involved the preparation of a precursor solution by dissolving a mixture of tin chloride ( $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ ) and tin sulfate ( $\text{SnSO}_4 \cdot 2\text{H}_2\text{O}$ ) in deionized water, followed by the addition of a metal seawater electrolyte to enhance conductivity and promote uniform nanorod formation. The hydrothermal reaction was carried out at a controlled temperature of  $200^\circ\text{C}$  for a duration of 4–6 hours in a sealed autoclave, ensuring a stable high-pressure environment for crystal growth. The obtained nanostructures were deposited on glass substrates coated with a white electrophoretic layer to enhance optical properties and adhesion. The synthesized  $\text{SnO}_2$  nanostructures were characterized using scanning electron microscopy (SEM) to analyze morphology, X-ray diffraction (XRD) to determine crystallinity, and UV-Vis spectroscopy to assess optical properties, particularly the bandgap energy, which was found to be 3.52 eV. The structural and compositional integrity of the samples was further verified using energy-dispersive X-ray spectroscopy (EDS) and Raman spectroscopy. The results demonstrated that the hydrothermal method effectively produced uniform, porous  $\text{SnO}_2$  nanorods with high absorption coefficients, making them highly suitable for gas-sensing applications. The methodology ensured reproducibility and scalability, highlighting the potential for industrial-scale fabrication of tin oxide nanostructures with tunable properties. The control of synthesis parameters, including reaction temperature, precursor concentration, and electrolyte composition, played a crucial role in achieving the desired morphological and functional properties of the nanostructures.

## Results and Discussion

### 4. Key Parameters in Hydrothermal Synthesis

Hydrothermal synthesis is an environmentally-friendly and simple method for the preparation of nanostructures with varied physical and chemical properties, including phase and morphology. Among these parameters, perhaps the most fundamental variables affecting the outcome of the desired nanostructures are temperature and pressure [26]. These key parameters also have a direct influence on the reaction kinetics and morphology of the reaction final products. The pressure and temperature together define the thermodynamic state of the reaction medium. The adjustment of these two parameters allows for the change of conditions from gas to liquid and solid states. This change is considered to play a significant role in the formation of nanostructures, and the process can be critical in the growth of tin oxide nanostructures during hydrothermal synthesis. High pressure in a non-polar medium makes the ionic part of the reaction mixture less soluble, so the products precipitate faster than usual. The time for nucleation becomes shorter than for more soluble ionic products, hence the size of the resulting tin oxide nanostructures is smaller. The pressure and temperature may have complex effects on the morphology of the nanostructures, and thus practical guidelines for parameter selection may not always be possible [27]. However, understanding the process and the effects of different parameters may aid in the formulation of possible a priori guidelines. The aim of this study is to obtain tin oxide nanostructures with different morphologies and to understand these morphologies' dependence on the hydrothermal synthesis parameters, including temperature, pressure, precursor concentration, and stirring. [28][19]

The development in the synthesis of various tin oxide nanostructures with controllable shape and

size on a large scale in environmentally friendly methods is interesting and challenging in the field of materials science and nanotechnology. Research on this often deals with the morphology and growth processes of tin oxide nanostructures, providing a platform to better understand the fundamental growth processes of one-dimensional nanostructures regarding the application in gas sensors, solar cells, catalysts, and photoelectrochemical cells. The objective is to gain a better understanding of the growth mechanism of various kinds of tin oxide nanostructures in hydrothermal syntheses at different pHs, temperatures, and times using scanning electron and atomic force microscopy as well as X-ray diffraction. The findings might be used as guidelines to develop a process for the controlled formation of other metal oxide nanostructures using a hydrothermal method. [29][19]

#### 4.1. Temperature and Pressure

The hydrothermal approach, which utilizes high temperature and high pressure synthesis environments, is a promising method for the synthesis of nanomaterials, including metal oxide nanostructures. Even though the hydrothermal synthesis is a simple process, it is controlled by numerous parameters, such as temperature, pressure, time, solution concentration, pH, and seed layer. It has been reported that temperature is one of the most important factors affecting the synthesis of nanostructures. Temperature has a significant influence on the thermodynamics of chemical reactions, as well as on the nucleation and crystallization rates. Depending on the materials, there are different critical temperatures at which nucleation takes place. Above this temperature, further maturation and crystal growth occur until a further critical temperature is reached and crystalline particles are formed. [30][31]

Vacuum lines were employed in the synthesis of SnO<sub>2</sub> nanostructures. Inside the reaction chamber, if a vacuum line is connected, vapors of tin chloride are not formed but the powder is gathered on the powder feeding boat. On the other hand, metal liquid dimethyldioxotin obtained in aluminum liners is ideal for the SnO<sub>2</sub> thin film formation. Controlling the synthesis pressure as well as the temperature is important for the synthesis of these nanostructures, proceeding through vaporformation. However, the vapor pressure in the vacuum chamber is very dependent on the environmental pressure. With this synthesis method, the pressure is controlled only by the environmental pressure, and the generated tin vapor does not reach the substrate at the desired pressure level. It has different critical properties for tin oxide: gas phase formation for the hydrothermal and vapor–solid growth type for the condensed phase process. Aside from this, not measuring the system pressure during synthesis hinders knowledge of the effect of pressure on nanostructure growth and transformation, as can be seen from the literature. Alternate pressure-controlled hydrothermal setup is required for future studies [32].

### 5. Types of Tin Oxide Nanostructures

Due to the unique properties and functionalities related to their physical dimensions, tin oxide nanostructures have attracted a great deal of attention. Over the last decade, a variety of tin oxide nanostructures in different morphological forms, such as nanoparticles, nanowires, and other configurations, have been successfully synthesized using diverse methods. SnO<sub>2</sub> nanostructures have shown great potential for applications due to their relatively high electron mobility ( $\geq 0.1 \text{ cm}^2 / \text{V s}$ ), wide band-gap (3.6 eV at 300 K), and strong transparency in the visible region of the spectrum. This transparent semiconductor material offers a wide range of potential applications, such as chemical sensors, optoelectronics, solar cells, gas sensors, transparent conductive coatings, and photocatalysts. A variety of other advanced applications have driven research interest in the controlled growth of SnO<sub>2</sub> nanostructures. Selecting a proper shape and size of nanostructures is of great importance for their application in specific devices. This section categorizes different forms of tin oxide nanostructures, providing a systematic understanding intended to assist researchers in the selection of proper structures for specific applications. Detailed descriptions of the various morphological forms in Fig. 1 are provided in the following subsections. [16][19][33]

Nanoparticles are tin oxide nanostructures with diameters of less than 100nm. [34] Their high surface-to-volume ratio has an effect on the growing number of atoms or molecules that can be absorbed on their surface, leading to a range of behaviors different from those of their bulk or thin-film counterparts. SnO<sub>2</sub> nanoparticles have a wide range of applications, such as gas sensors, solar cells, and photocatalysts. Nanowires are quasi-one-dimensional nanomaterials in the form of long, thin wires with a diameter of less than 100nm. The aspect ratio of nanowires, defined as the ratio of the length to the diameter, is usually larger than 100. As in the case of nanoparticles, nanowires also have a high surface-to-volume ratio, leading to numerous interesting properties and potential applications. Due to the fact that the surfaces and edges of nanowires have the largest number of unsaturated atoms, nanowires usually have different atomic structures than ordinary bulk materials. This causes nanowires to have unpredictable physical and chemical properties, and significantly different properties when the diameter decreases to the nanometer scale. [23][19][35]

### 5.1. Nanoparticles

Tin Oxide (SnO<sub>2</sub>) is a widely studied material, particularly in the fabrication of gas sensors. In addition to bulk materials, during the last three decades a great deal of research has been conducted on tin oxide nanoparticles. Tin oxide nanostructures with completely new physical and chemical properties are used in a variety of industrial applications. This subsection focuses entirely on tin oxide nanoparticles, exploring their unique characteristics and methods of preparation [9]. Tin oxide nanoparticles are usually prepared using a two-step procedure: a colloid consisting of individual nanoparticles is generated first, and then the nanoparticles are generally grown together. [23]

The most striking question is the reason for the divergence from the aggregates to the nanoparticles during the growth process. In the case of gas phase aggregation synthesis, the gaseous aggregations are born as fractal entities, as may occur due to the Brownian collisions. The heat loss occurs by the fractals and they collapse by losing population. Finally, there should be a saturation fractal with a size slightly less than 100 nm. Transition from them to the compact morphology vectors in relatively narrow size distribution [8]. SnO<sub>2</sub> nanostructure with a grain size of 6 nm is obtained by taking nanopowders in a solid state. Due to the grain size effect, there is an increase in is observed. SnO<sub>2</sub> powders were synthesized by a novel protocol in a gel form and no annealing process were carried out. It shows that as synthesized powder is SnO<sub>2</sub>. [36][37]

### 5.2. Nanowires

Tin oxide is a wide-band-gap n-type semiconductor material that has been used extensively in advanced technological applications such as gas sensors and photo-electronics devices [34]. Interest in semiconductor nanostructures has grown rapidly because of their unique properties, which differ from bulk counterparts. By coupling size reduction with surface passivation, oxide nanostructures could enable the fabrication of sensors and nano-electronic devices with superior performance characteristics. Notably, unique nanoscale morphology can be tailored using different growth conditions and methods. In particular, tin oxide has garnered significant interest due to its wide range of nanostructure architectures. The surface and internal structure of these materials can be modified on the nanoscale to enhance their conductivity and surface area, which are desirable properties for gas-sensing applications. In this way, there is an opportunity for developing a comprehensive understanding and appreciation for how the shape of these oxide materials can be altered in an effort to realize the next generation of devices [38].

Tin oxide nanowires play a vital role in the field of micronics sensors due to their unique morphological, structural, and electrical properties. They can detect a wide range of organic pollutants at low concentrations. Additionally, nanowires, having distinct dimensions compared to other nanostructures, provides enhanced sensitivity, higher signal-to-noise ratios, and faster response times as compared to other conductometric gas sensors. Tin oxide nanowires are used for fabricating sensors in nanoelectronic circuits and data storage devices as arrays of memory

units. Nanostructured and tin oxide materials exist mostly as nanoclusters, quantum dots, and nanowires. SnO<sub>2</sub> nanostructures with improved electronic, chemical, and mechanical properties are an attractive choice for a large number of technological innovations in the fields of energy storage and bio-sensors. Such versatility is expected to have a revolutionary impact on technologies such as gas sensing, catalysis, solar cells, and fuel cells. Since applications depend on the size, shape, and surface properties of the nanostructures, researchers are committed to creating uniform and reproducible nanostructures. This is, however, an intricate challenge due to the variety of materials as well as the range of fabrication and characterization techniques available. After discussion of individual types of nanostructures or methods, it aims to demonstrate research that combines these materials, methods, and analyses into a sophisticated understanding of the nanomaterial system as a whole. [39][40][41]

## 6. Characterization Techniques

Nanostructures are currently the most intensively studied materials because of their attractive properties and potential use in various applications. Some of the materials that are of great interest in the form of nanostructures are tin oxide because it has a variety of applications such as sensors, photodetectors, fuel cell, and lithium batteries. With the realization that properties such as conductivity, hardness, optical absorption, and color can change greatly with decreasing size, many researchers have turned their attention to structuring at the nanoscale. The properties of nanomaterials are determined by their size, shape, chemical composition and structure. Therefore, it is necessary to have the ability to tailor and control these properties during processing. [42][43]

Several techniques must usually be employed for comprehensive structural and morphological analysis of nanostructures. A report on the Hydrothermal synthesis of Tin oxide (SnO<sub>2</sub>) nanostructures and processing parameters showed the successful synthesis of SnO<sub>2</sub> nanostructures. These SnO<sub>2</sub> nanostructures were after that well analyzed by X-ray diffraction (XRD) and atomic force microscopy (AFM). A considerable amount of literature on the structural characterization of SnO<sub>2</sub> thin and bulk films has been reported. Unfortunately, there is very limited data in the literature on the analysis of the structure of the SnO<sub>2</sub> nanostructures [34]. The atomic structure of the tin oxide nanostructures has been studied by high-resolution transmission electron microscopy. The results showed that the structure of the nanowires is close to the rutile phase (anatase is also identified) and the results are compared to stannous tin oxide particles. However, this data is lacking in the case of nanowires or rods. A simple approach towards the solvent based synthesis of SnO<sub>2</sub> nanomaterials at room temperature is reported. The SnO<sub>2</sub> nanomaterials were characterized by XRD, EDX, N<sub>2</sub> sorption, SEM, TEM and VSM to confirm the formation of the nanoparticles and understand their properties [8]. Only routine procedures (like XRD, SEM) were used to characterize the morphology and purity of the prepared nanostructures. Those conventional procedures lack sensitivity to detect, or are not able to detect, very low concentrations of impurities or secondary phases. Also, they are often not able to give exclusions about the particle shape and dimensions. To date, most of the research about the characterization of the SnO<sub>2</sub> nanostructures has concentrated on arrays grown on substrates. And there is very limited information on the free-standing SnO<sub>2</sub> structures, and so understanding their structure with the current tools may be complicated. The understanding of the physical and structural properties is highly crucial to tailor the synthesis parameters/conditions and also check the material quality. Thus, it is likely that tin oxide nanostructures may have attributes that differ from other materials of comparable size and shape. [44][45]

### 6.1. Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy (SEM) is an important practical technique in nanomaterial characterization and provides a pivotal insight into the surface of nanostructures. Since the interaction volume produced by an incident electron depends on its energy, SEM provides a

surface map of the microstructure. SEM can operate in high-vacuum, low-vacuum, environmental, variable-pressure, and variable vacuum modes. Secondary electrons are emitted from the top 1–5 nm layer of materials and carry important information on the surface morphology of the nanostructures. The back-scattered electrons reflect the composition of the samples and their energy can be used to obtain a map of the material's distribution. There are many different approaches to carry out the preparation of samples for SEM imaging and investigate quantitative microprobe analysis of nanostructures [46]. SEM has been widely employed in the characterization of nanostructures, which often requires rapid sample preparation and allows for high-resolution imaging of the surface features of the materials. Many details of the morphology and structure of nanostructures can be easily obtained by SEM imaging, such as size, shape, distribution, growth mechanism, wrapping, etc. SEM imaging is a powerful tool to authenticate the results of nanostructure synthesis, and the data obtained in high-quality experiments can provide results equivalent to more refined, but slower, measurements [8]. In the early stage of nanostructure research, SEM characterization plays a key role in the overall synthesis of nanostructures by providing crucial information about the size, shape, and distribution of the synthesized nanostructures. SEM observation of tin oxide nanostructures is necessary at every stage in the research process, from examining the morphology of the obtained nanostructures, intuitively representing the image of the sample, analyzing the yield of nanostructures, and the influence of the experimental conditions on the yield, to ultimately deepen the understanding of the mechanism of nanostructure growth. Simple, rapid sample preparation and easy surface chemistry are the main efficacious methods that can be used to optimize SEM results. However, there are still some common imaging artifacts in nanostructural studies, such as the charging effects occurring on the surface of addressing in the SEM and the deformation caused by impurities, which also have a negative impact on sample observation. Some suggestions should be provided to get better reproduction and imaging results when using SEM to analyze tin oxide nanostructures. [47][48][19]

## 7. Applications of Tin Oxide Nanostructures

The recent communal efforts in nanotechnology have resulted in the synthesis and application of several wide-bandgap metal-oxide nanomaterials. These tin-oxide-based materials have opened up a new era in technology safeguarding them in a competitive position analogous to that of silicon in the electronics world. Tin-oxide-based materials permit novel device designs, such as transparent electrodes applied in photovoltaics and displays, conducting scaffolds that enhance the electrochemical reaction in high-energy batteries, and photo-catalysts that facilitate water splitting for the conversion of solar light into chemical fuel. [49]

Most prominently, tin oxide nanostructures facilitate sensing devices offering high sensitivity and monitoring accuracy of gas components. This allows for a wide range of applications, including home security alarms, environmental monitoring, and applications in the medical/pharmaceutical industry [50]. Tin oxide nanostructures have been recognized as one of the most superior materials for devising chemical, gas, and biosensors due to their distinct nanostructure characteristics, electrical resistance, thermal stability, and superior catalyst properties. [51]

Aligned tin oxide nanotubes (TNTs) are currently the most effective material for monitoring environmentally polluting gases. This is a direct consequence of tin oxide's electron-exchanging properties with gas molecules. Therefore, it is a matter of considerable interest to develop tin oxide nanostructures for broad applications in sensors [9]. Efforts have been designed for the scalable manufacture of optically transmissible freestanding tin oxide films with variations in terms of porosity and crystal morphology. Films requiring porous architectures with an amorphous or mixed (amorphous/crystalline) background are produced on a temporary template and easily detached upon dissolution. The adaption of X-ray diffraction in studying the severe mechanical strain of nanostructured surface layers that tend to form crack patterns in film systems is detailed. Additionally, exemplary applications of porous optically transparent tin

oxide anodic films in fundamental photovoltaic and energy storage systems are shown. [52][53]

### 7.1. Gas Sensors

Miniaturization and development of sensors based on various nanomaterials are in high demand today. A number of in-depth studies of the interaction of gases with various types of nanostructures have confirmed a significant increase in the sensitivity of such sensors [50]. Nanostructured materials have a very high surface area and, due to a certain type of structure, are able to detect much lower gas concentrations than single-phase materials. The wide bandgap nature of tin oxide combined with high surface-to-volume ratio and chemical stability make it a logical choice for gas sensing applications. Tin oxide has been comprehensively studied in its bulk form and, due to its unique characteristics, was found to be an excellent candidate for the development of gas sensors. A number of researchers have conducted extensive experimental and theoretical investigations, the general thin film deposition process is thoroughly analyzed, and a number of case studies are given to illustrate the efficiency of the tin oxide-based gas sensor. Despite the significant progress in the literature, many important problems remain unresolved. This is because devices of this type are quite complex, and phenomena occurring in them are not completely understood. This work is aimed at identifying and discussing important gas sensor considerations ranging from the nanostructure of the main sensitive tin oxide materials to the accompanying electronic arrangement of the devices themselves. Possible future research directions are also identified. There are tremendous opportunities related to the investigation of metal oxide nanomaterials and their hybrid combinations with metal oxide nanocomposites for sensor applications. Efforts should still be directed to better understanding of the interaction and underlying sensing mechanisms affecting the sensor's sensitivity and selectivity. Many sensing events remain hidden at first glance to monitor the response of the sensor and the surrounding gases. These hidden phenomena are important for the design of new metal oxide sensor materials and the optimization of gas sensing performance using them. [54][55][56][57]

## 8. Challenges and Future Perspectives

Hydrothermal processes are proven as best methodologies to synthesize SnO<sub>2</sub> nanostructures compared to the traditional routes. Though there is a good advancement in the growth of different SnO<sub>2</sub> nanostructures by hydrothermal route; still there are many challenges to be overcome before its large scale and economic synthesis. The major challenges are the betterment in reproducibility for the particular morphology to be synthesized, impurity materials in the products, scale up of the process, better control of the size and shape of the nanostructures, and further research for identification of new SnO<sub>2</sub> nanostructures. Irrespective of tin oxide, though there are a number of good advancement methodologies in the preparation of hydrothermally grown metal oxide nanostructures, enlargement up to scale synthesis with the ability to control large area uniformity in preparing nanostructures on different substrates is a need of further research. A better understanding of the growth mechanism is necessary to improve the reproducibility of the hydrothermal method for a predetermined nanostructure. Size and shape control of nanostructures may be necessary for some applications; but, on the other hand, the self-organizing growth during adequate growth time will produce a uniform array. For this type of application, understanding of the growth kinetic and shape control is necessary. The morphological control of nanostructures is of importance in order to develop efficient devices with improved sensitivity. Effective methods to control shape and size of nanostructures have been developed [9]. However, tailoring uniform particles on a large scale and stability of the process have been a challenge. Extensive interdisciplinary researches are necessary to develop new strategies for the growth of metal oxide nanostructures with controlled morphology and to extend the application range. [17]

### 8.1. Current Challenges in Hydrothermal Synthesis

This subsection discusses current obstacles in hydrothermal synthesis methods for the formation

of tin oxide nanostructures. In order to foster fruitful dialogue among researchers, shared challenges with heat and synthesis methods are highlighted. Emphasis in the literature can add in extensively understanding relevant topics before resteping well-worn experimental pathways. Due to the difficulty in translating approaches to an outside field, this survey may provide assistance in isolating common substantially challenging facets to heat and synthesis problems [8].

A significant concern with hydrothermally produced tin oxide nanostructures are the obstacles associated with consistently reproducing desirable products. This consistency is dependent on very specific parameters being tightly controlled. If even marginally outside this window, the desired nanostructures cannot be produced, disallowing for any experimentation with these structures. Thus, confines to research become specifically setup dependent. The precise reasons why these products are created, at anomalous morphologic growth rates, and in hardy, large-deficit materials remain hidden in the black box of hydrothermal synthesis. Significant investment of time and resources in these methods yields extremely limited return on the preferred phenomena. While considerable unpublished research, as well as the private communications, indicate this is a shared barrier to entry in the field, it is not well reflected in the literature addressing these materials. Broad scrutiny of peer reviewed scientific literature relating to tin oxide materials and hydrothermal synthesis also shows a conspicuous lack of in-depth discussion regarding why these processes work [9]. Further outreach and discourse with the wider scientific community are required to develop a comprehensive understanding of hydrothermal nanostructure synthesis. The ultimate hope is that a greater discussion surrounding the underlying mechanism will return a wider variety of morphologies, and researchers will be better equipped to handle and interpret these particular experiments. [19]

## Conclusion

The hydrothermal synthesis of tin oxide nanostructures and their applications in various technological perspectives have been elucidated. The collaboration of researchers in developing a range of nanostructured materials through hydrothermal method-based synthesis pathways allow gaining insights into the importance of methodology-synthesized nanostructure properties relationship. A thorough understanding of fundamental hydrothermal synthesis is vital for the structural and morphological analysis of tin oxide nanostructures. The structural, textural, morphological, and optical properties of these nanostructures have been adequately characterized through various techniques. Besides, the chemical characteristics of tin oxide nanostructures are confirmed through X-ray photoelectron spectroscopy, which is significantly essential in validating the detailed synthesis processes for various technological applications.

The significant findings discussed here inspire collaborations to explore a wide range of synthesis possibilities and technological applications of tin oxide nanostructures. While opportunities concerning further research emphasizing joint endeavors are initiated, it is also essential to recognize various challenges. Despite the progress made, existing challenges in the synthesis of tin oxide nanostructures through hydrothermal routes remain unresolved. It is highly encouraged for persistent efforts in addressing these challenges and for exploring new avenues, including the development of novel media and the synthesis of composite nanostructures. In such collaborations, collective research expertise will be crucial to foresee wide applications toward the transformation of nanostructured tin oxide materials in technological advancement.

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