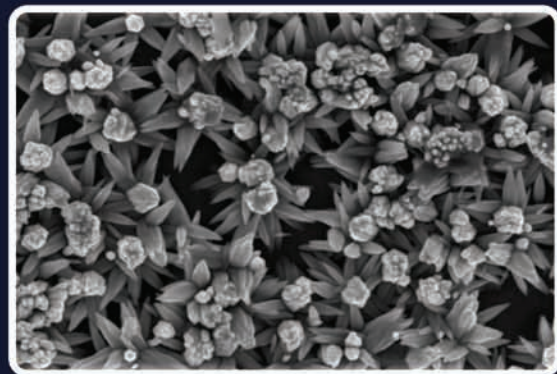
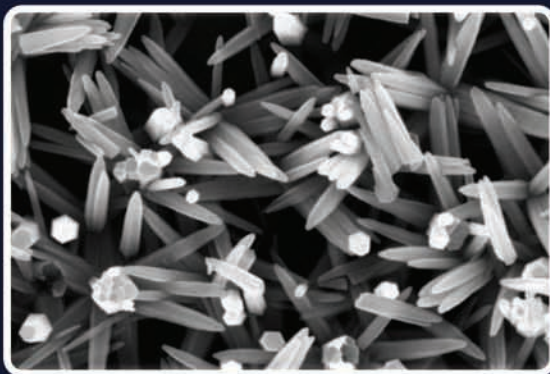
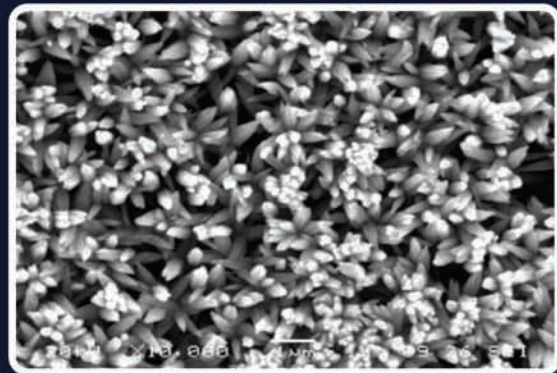
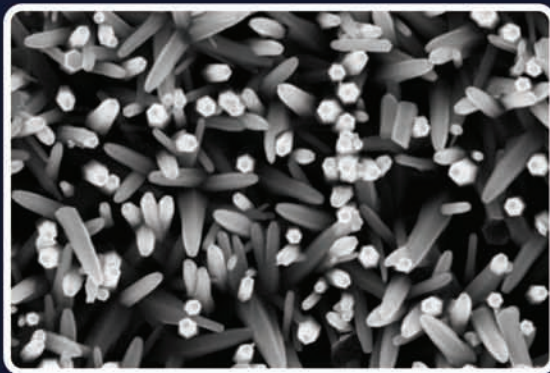


# THIN FILM NANOMATERIALS: SYNTHESIS, PROPERTIES AND INNOVATIVE ENERGY APPLICATIONS



Editors:

**Sampat G. Deshmukh**

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**Kailash J. Karande**

**Swanand G. Kulkarni**

**Bentham Books**

# **Thin Film Nanomaterials: Synthesis, Properties and Innovative Energy Applications**

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## FOREWORD

A thin film is defined as a low-dimensional material created by condensing, one-by-one, atomic/molecular/ionic species of matter. It is a layer of material ranging from monolayers to several micrometers in thickness. The dimensions of thin films are in the same range as nanomaterials. Thin films have properties that can be different from those of their corresponding bulk structures. The films have different surface properties than their bulk behaviour. Thin films have a larger surface-to-volume ratio, hence the surface and near-surface characteristics decide the properties of the thin film. As a result, thin film properties depend on the thickness of the film, substrate nature, and deposition method used for the fabrication of thin films. Thin film fabrications are generally carried out by depositing the required material in the atomistic deposition (atom by atom) over the substrate, which may result in either a single crystalline, polycrystalline, or amorphous structure depending on the deposition conditions.

Thin film has the potential to engineer various properties such as porosity, surface morphology, surface roughness, and crystallite size. These advantages assist in the development of new products and minimize wastage compared to conventional manufacturing techniques. This book provides an overview of various thin film processing methods, mechanism behind the growth, and tools used for the characterization of thin films.

Thin-film technologies are prevalent in virtually every industrial sector. For example, this technology is essential for fabricating active and passive devices, complex optical systems, and even protective coatings for tools. Thin film applications include thin film batteries, optics, magnetics, resistors, solar cells, and many more.

Complex nanomaterials thin films are finding applications in a wide range of energy and information technologies, including electron and ion conductors, photovoltaics, thermoelectric, dielectrics, and resistive switching. Irrespective of whether single thin films or more sophisticated multilayered systems are employed, each of these technologies requires a thorough knowledge of the thin film's physicochemical properties, its interactions with the environment, and the role of defect/interface/boundary effects that might stem between film and substrate, layers with different compositions or even simply from the grain boundaries. Although it depends on the technology area, the key properties might be different while many of the materials optimization strategies may be common.

The overall aim of the collection of materials is to spark extensive discussions of the advances that materials physics and chemistry can make in thin film applications.

In this book, significant papers on synthesis, characterizations and applications of some nanocrystalline films for versatile applications are collected. The book is divided into eleven chapters and covers a variety of nanomaterials including metal oxides, sulfides and selenides, polymers, and carbon-based materials. Thin films of TiO<sub>2</sub>, ZnO, Fe<sub>2</sub>O<sub>3</sub>, and other oxide films are used in different applications including photocatalytic degradation, Li-ion battery, *etc.* Similarly, quaternary compounds like Ni-Cu-Fe-O are applied for gas sensing. The compounds like Cu(InGa)Se<sub>2</sub>, CdS, ZnS, MoS<sub>2</sub>, *etc.* are used in photovoltaic applications.

The book should be a welcome addition to the much-needed analytical studies on various applications. I believe that the authors and editors of the book are responding positively to globalization and environmental concerns by prompting friendly energy sources, and storage mechanisms and sensing essential approaches to appreciate the diversity and complexity of nanotechnology.

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## PREFACE

Progress in nanomaterials has led to the development of mankind. The visionary statement given by Richard Feynman ‘**There’s Plenty of Room at the Bottom**’ has been experienced within 64 years by the scientific community across the world. The research fraternity has made continual efforts and noteworthy contributions in the field of nanotechnology.

Nowadays, research on thin film nanomaterials is rapidly developing to lead to minimizing the device dimensions. Thin-film nanomaterials have diverse applications in many fields *viz.* optoelectronic devices, energy generation and storage, gas sensors, medical, defense, textile, *etc.* Moreover, with respect to the increasing human population and industrialization, the energy demand is continuously increasing. For the improvement of the energy efficiency of the current devices, the incorporation of thin film nanomaterials becomes essential.

There are a lot of books that focus on a particular area of energy or the ideologies of deposition. However, the current technology needs the overall scenario in the development of nanomaterials. The present book titled ‘**Thin Film Nanomaterials: Synthesis, Properties and Innovative Energy Applications**’ demonstrates an overview of the synthesis, properties, and innovative applications of thin-film nanomaterials. This book also provides an overview of the employment of thin film nanomaterials over a wide range of energy applications.

In the first chapter, the synthesis and characterizations of cadmium and zinc sulphide thin films are elaborated for opto-electronics energy devices. The critical review of CIGS thin film nanomaterials from synthesis to recent energy applications has been discussed in the second chapter. From the deposition to advanced applications like sensors, supercapacitors and solar cells of metal oxide nanocomposite films are elaborated in chapter three. Nanostructured TiO<sub>2</sub>@carbon films deposition *via* CBD for photocatalytic applications is discussed in the fourth chapter. The fifth chapter describes the bandgap engineering along-with optical properties of filler reinforced PMMA composite films with current challenges and future perspectives in material science and technology. The synthesis, recent developments and advanced applications of metal oxide thin films are discussed in sixth and seventh chapter. Chapter eight gives information about the synthesis of iron oxide films *via* the chemical route and its characteristics for solar cell and green energy storage applications. The tenth chapter elaborate the challenges and future prospectus of CNT-based

cathode emitters for various applications. In the last chapter, the advanced characterizations of nanocrystalline ferrimagnetic thin films deposited *via* spray pyrolysis technique for sensor energy are elaborated.

It is a great honour for us that Prof. Chandrakant D. Lokhande, who ranked First in India as a researcher in the field of Allied Physics according to Stanford University, United State, has written the foreword.

Lastly, the editors wish to express their sincere gratitude to the authors and peer reviewers. We are also very grateful to our publisher Bentham Science for providing an opportunity to publish this work. One of the editors (SGD) expresses his gratitude to his mother Smt. Vimal (Amarapur-Deshmukh Mala) for her continuous blessings to reach this milestone. We expect this book will be beneficial to professors, scientists, engineers, research scholars, postdocs as well as UG/PG students.

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**CHAPTER 1****Synthesis and Characterization of CdS and ZnS Nanostructured Thin Films for Opto-electronic Energy Applications****Raghavendra Bairy<sup>1,\*</sup>, Vijeth H.<sup>2</sup>, Rajesh K.<sup>3</sup> and Rohan S. Deshmukh<sup>4</sup>**

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**Abstract:** Since their early discovery, thin films have quickly found industrial uses, including decorative, optical, and energy storage applications. The range of applications for thin film technology has expanded to the point where nearly every industrial sector now uses it to impart specific physical and chemical properties to the surface of bulk materials. The ability to customize film properties by varying the microstructure through the deposition parameters used in a particular deposition technique has recently allowed them to advance from the most basic applications, like protective coatings against wear and corrosion, to the most technologically advanced ones, like microelectronics and biomedicine. Despite such remarkable advancements, the relationship between all phases of the fabrication of metal sulphide thin films such as CdS and ZnS specifically deposition parameters – morphology and characteristics, is not entirely precise. In summary, the characterization of thin films involved several techniques, including X-ray diffraction, UV-Vis spectrophotometry, scanning electron microscopy, energy-dispersive X-ray diffraction, and transmission electron microscopy. The investigation of nonlinear optical (NLO) parameters was carried out through open aperture (OA) and closed aperture (CA) Z-scan measurements, employing a diode-pumped solid-state continuous-wave laser at 532 nm excitation. The NLO parameters, namely the nonlinear absorption coefficient ( $\beta$ ), nonlinear refractive index ( $n_2$ ), and third-order NLO susceptibility ( $\chi^{(3)}$ ) exhibited an increasing trend with higher doping concentrations. These promising outcomes regarding the NLO parameters in nanostructured CdS and ZnS thin films with increasing doping concentrations suggest that these processed films hold significant potential for applications in opto-electronic energy-related technologies.

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**Keywords:** Characterizations, Morphology, NLO, Properties, Structure, Thin Films, Z-scan.

## INTRODUCTION

CdS semiconductor thin films are widely utilized in solar cells, lasers, photoconductors, light-emitting diodes, opto-electronics, and photonics, among other devices [1-4]. A lot of research has been done on sulfides (S) of the semi-conductive chalcogenide nanoparticles group II-VI because of their intriguing opto-electronic capabilities [5]. The capacity to dope (metals) CdS with suitable impurities allows enhancement of its varied properties for advanced applications. The band gap of CdS, an n-type semiconductor with a simple cubic structure and a high absorption coefficient, is almost 2.42 eV [6]. In recent years, the scientific field of NLO has experienced rapid expansion. It is based on the phenomenon known as spectacle, which occurs when a material interfaces with extremely coherent electromagnetic radiation. The transition of metal-doped semiconductors has received a lot of scientific and technological attention recently [7] because of their potential applications. As NLO materials, a variety of 'inorganic semiconductors' are employed. These materials have important applications in optical information processing and integrated optics and play a vital role in the field of NLO [8, 9]. As an outcome of thin films' potential applications in the arena of "science and technology" transition novel doped metal semiconductors have fascinated extensive research considerations in the modern years. In our society, cadmium is always present in useful products or in controlled waste. Cadmium chalcogenides were extensively investigated in both the nanoscale and the bulk. Due to their high light sensitivity and quantum efficiency, these materials are recognized for photovoltaics with great promise. In addition, the range of their direct band gap indicates the possibility of producing opto-electronic devices that respond to various electromagnetic radiations.

CdS semiconductor nanocrystalline thin films received increasing focus because of the enhanced NLO features [10]. The doping concentrations of various contaminants are the desired properties and convenience of thin CdS film material for current devices [11]. Recent years have seen a great deal of research into the CdS chalcogenide (ternary) thin films using a variety of processes, including the sol-gel SC approach [12], sputtering [13], SILAR [14], CBD [15], and SP technique [16]. For the large area of high-quality thin films, SP is different, appropriate, and a good deposition process. Doping CdS thin films with different metallic ions may allow one to modify their opto-electronic characteristics [17].

The literature contains far fewer reports on the "NLO properties of nanocrystalline metal-doped CdS semiconductor" thin films, whereas the 2PA has been extensively studied [18]. The application of spin-coated cadmium sulphide doped nickel thin films in the third harmonic is documented in the literature [19]. Due to the flexible NLO of the characteristic films, it can be employed to deal with specific needs. Scientists now regard it as very motivating and compelling with the intention of using it in NLO gadgets. Film quality was shown to be enhanced by dopant ions with lower radii than  $\text{Cd}^{2+}$  (0.097 nm) [20]. Using the SP process, high-quality undoped CdS and metal-doped films, such as Al, were created by integrating Al in various compositions, including 0%, 1%, 5%, and 10% by weight. To develop the sign and magnitude of the third-order NLO characteristics, a continuous wave laser (with an output power of 200 mW) was used as the excitation source by the Z-scan technique. Numerous applications make heavy use of CW lasers in the mW to kW range [21].

ZnS is a direct transition II-VI semiconductor material with a large direct band gap that is extremely important in many applicable fields of science and technology [22]. The use of this material in light-emitting and opto-electronic applications, including solar cells, optical coating, and electroluminescent devices, is quite promising [23]. It is regarded as a highly promising material for applications in blue light-emitting laser diodes and thin-film electroluminescent displays [24]. The growing need for innovative nonlinear optical materials with potential applications in integrated optics has prompted many studies into wide-band gap semiconducting materials in recent years [25-27]. Long-term research has also been performed on nonlinear optical responses in relation to the use of photonic devices and laser operation [28]. Usually, nonlinearity is seen at very high highlight intensities, such as those produced by lasers. Exceptional photophysical characteristics and significant optical nonlinearities include optical logic gates, optical switches and limiters, fast optical communications, *etc.* [29]. There are several methods being explored for thin film preparation.

Chemical Spray Pyrolysis (CSP) is the most affordable and straightforward technology, and it is utilised for large-scale industrial manufacturing [30]. The CSP enables spraying of the precursor solution onto the heated substrate. The heat of the substrate disintegrates the solution's molecules into their constituent parts. The crystalline characteristics and surface morphology of the substrate are highly dependent on its temperature. The substrate temperature may be changed from 100°C to 500°C using the CSP process. The kind of substrate, composition, solution flow rate, the time required for deposition, and distance from the nozzle tip to the substrate utilised, all affect the quality of the deposited film. The films prepared are

## Thin-Film Photovoltaics Using Cu(In,Ga)Se<sub>2</sub> Nanomaterials

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**Abstract:** Cu(In,Ga)Se<sub>2</sub> (CIGS) is a promising absorber material for thin film solar cells because of its excellent thermo-chemical stability and high power conversion efficiency. Despite the excellent performance, commercialization of CIGS solar cell technology has been hindered due to issues related to the preparation of the absorber layer. The manufacturing of CIGS absorbers needs innovative technological development to make them commercially competitive, simplified and cost-effective. In this connection, the solution process utilizing CIGS nanomaterial precursor is a non-vacuum, low-cost, non-toxic and scalable approach with a high potential for developing an absorber layer. The typical processes comprise the synthesis of high-quality CIGS nanomaterials followed by printing constituent precursors in thin film form. Subsequently, thermal/photonic post-treatments of the printed precursors transform into a high-quality photovoltaic-grade absorber. The chapter critically reviews CIGS nanomaterial synthesis methods and discusses various printing techniques. The discussion follows an investigation of printed thin film's thermal and photonic processing to realize a high-quality CIGS absorber layer suitable for thin film photovoltaics. The processing parameters such as annealing profile, post-treatment, annealing atmosphere, Selenium source, photonic fluences, and alkali doping are discussed to understand their impact on the absorber's composition, morphology, and optoelectronic properties. The findings and related reviews afford critical insight into the absorber thin film design to improve the performance of solution-processed chalcopyrite solar cells. Finally, current challenges and prospects for effective technology implementation are discussed.

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**Keywords:** Alkali doping, CIGS, Intense pulsed light, Laser annealing, Nanomaterial, Photonic sintering, Selenization, Solar cells, Thin films.

## INTRODUCTION

A usable form of energy is inevitable for the continuous operation of humankind in the modern world. Solar energy-based resources are expected to gratify the growing demand for clean energy in the present and future, as anticipated by the International Energy Agency [1], minimizing the impact of global warming while reducing dependence on fossil fuels. However, the major challenge is to harness solar energy into electrical energy in an environmentally benign and economically viable way. In this connection, photovoltaic technologies must address interrelated constraints of power-conversion efficiency, stability (over 20-25 years), cost (comparable to fossil fuels), material abundance, toxicity, and recycling. Amongst all the solar photovoltaic technologies, silicon-based solar cells are leading with power conversion efficiencies [2], exceeding 25% on lab scales and producing more than 20% module efficiencies. Crystalline Silicon-based solar technology significantly contributes to photovoltaic-based power plants, with a market share of more than 90% [3]. Crystalline silicon solar cells usually have an absorber wafer of more than 100  $\mu\text{m}$  thick, making it difficult for decorative and building integrated photovoltaic applications. Thin film-based solar cell technologies have been considered cost-effective and supplementary to crystalline Silicon-based technologies. Thin film solar cell (TFSC) technology [4] typically employs absorber materials with a high absorption coefficient of  $10^5 \text{ cm}^{-1}$ . As a result, a thin film ( $< 5\mu\text{m}$ ) is sufficient to absorb maximum light falling on it; therefore, it is expected to have low manufacturing cost as the quantity of material utilized is very low compared to that of silicon cells. Apart from 1<sup>st</sup> generation Silicon-based solar technologies, prominent 2<sup>nd</sup> generation thin film technologies comprised of CdTe, Cu(In,Ga)Se<sub>2</sub> (CIGS) and amorphous Silicon solar cells have exhibited high potential. The 3<sup>rd</sup> generation photovoltaic technology, such as hybrid perovskite solar cells, has also demonstrated high power conversion efficiencies [2] exceeding 20% but suffers from poor stability, has a poor life for power generation, and is in the R&D stage. The second generation solar technology has demonstrated substantial improvement over the years. Despite higher toxic Cd content, CdTe technology has grabbed about 6% of the global market share through simple processing [3]. A power conversion efficiency of about 13.6 % has been reported for a-Si solar cells [2]; therefore, they have limited use in portable devices like a calculator. CIGS solar cells are leading among TFSC technologies, with the highest power conversion efficiency of 23.35% on a lab scale and about 17% for modules [2]. CIGS has excellent optoelectronic properties and tunable band gaps with proven

technological benefits such as longer thermal and chemical stability in outdoor tests. CIGS thin film solar cell has tremendous potential to explore as building integrated photovoltaic materials. CIGS is a direct band gap material and belongs to a semiconducting group of I-III-VI<sub>2</sub> compounds. The tetragonal chalcopyrite structure of CIGS can be derived from cubic zinc blende structure like ZnSe by replacing Zn sites alternately with Cu and In. Each Cu and In atom has four bonds with Se, while each Se atom has two bonds with each Cu and In. In and Ga do not form an alloy; therefore, Ga replaces the In atom in CIGS with Ga alloying. As the strength of the Cu–Se, In–Se, and Ga–Se bonds differs, the lattice parameter ratio  $c/a$  is not two and varies from 1.96 to 2.01. As stated earlier, Ga substitutes In, in chalcopyrite structure, the band gap of CIGS material can be tuned from 1.07–1.67 eV based on the  $Ga/(In+Ga)$  ratio [5,6]. This property can fine-tune the material's band gap and obtain the desired band gap grading for high-performance solar cells. The first solar photovoltaic device based on chalcopyrite  $CuInSe_2$  (CIS) was reported by Wagner *et al.* [7] in 1970. Since then, CIS technology has undergone many modifications to obtain today's highly efficient CIGS TFSC. Typical high-efficiency solar cell (>20 %) has multilayer stacks of Al:ZnO/i-ZnO/CdS/CIGS/Mo/SLG substrate device structure [8]. Soda Lime glass (SLG) substrate has been a popular substrate material [9] as it is stable up to the temperature of 550 °C and is a source of an alkali like Na, which plays a significant role in the growth and crystallization of the CIGS absorber layer. A Molybdenum (Mo) electrode of 500–800 nm thickness is the preferred back contact material over other conducting materials. It does not alloy with Cu and has a matching thermal expansion coefficient with SLG and CIGS [10, 11]. A CIGS layer of 1.5–2 μm is enough to absorb visible light, and various physical and chemical routes can prepare the CIGS absorber layer. The chemical bath usually deposits the ultrathin 50 nm CdS buffer layer on the CIGS layer [12] to form a hetero-junction. Al:ZnO/i:ZnO is a popular front contact usually sputtered to obtain a 300–900 nm thick window layer [13, 14]. Ag or Ni/Al grids collect current from the cell. Co-evaporation and Sputtering selenization approaches are widely reported for fabricating the CIGS absorber layer. Co-evaporation [15] is the most widely adopted route to produce the highest efficiency in CIGS solar cells. The CIGS layer is grown at a substrate temperature of 450–550 °C under an ultra-high vacuum. The three-stage process is known to produce a band gap graded CIGS absorber layer with overall Cu poor ( $Cu < (In+Ga)$ ) composition. In the first stage, (In, Ga)-Se rich layer is deposited, followed by Cu-Se, and at the end again, (In, Ga)-Se layer is deposited, resulting in band gap grading at the front and back of the absorber layer. Typical power conversion efficiencies obtained by the three-stage process are more than 20%. Although highly efficient, the co-evaporation process is challenging to scale up uniformly in composition and stoichiometry on large areas. The process is complex,



## Thin Film Metal Oxide Nanocomposite: Synthesis to Innovative Applications *via* Chemical Route

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**Abstract:** Metal-oxide nanocomposites are promising in the fields of nanotechnology and nanoscience for a variety of application purposes, including sensors, supercapacitors, solar cells, etc. The increase in its practical application efficiencies may be due to these increased features. This chapter covers recent research on nanocomposites and their several possible uses. Additionally, metal oxide-based nanocomposite synthesis techniques are gaining popularity because they offer high production rates, high product yields, and minimal toxic waste formation while also being cost-effective and environmentally friendly. Physical and chemical methods have been used to synthesize metal oxide nanocomposites. This chapter provides an overview of the various chemical methods used to synthesize metal oxides. The many reported synthesis methods and prospective applications like solar cells, gas sensors, and supercapacitors of metal oxide-based nanocomposites are discussed in this research.

**Keywords:** Chemical methods, Nanocomposites, Sensors, Supercapacitors, Solar cells.

### INTRODUCTION

Nanotechnology is a branch of science that describes materials at dimensions less than 100 nm. Here, compared to similar bulk materials, particle sizes between 1 and 100 nm exhibit dramatically different chemical and physical properties, presenting

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both opportunities and challenges. Numerous environmental, energy, health, and other challenges may be affected by the employment of nanoscience and nanotechnology in sensing, anti-corrosive coating, energy storage, *etc.*, [1-6]. The novelty of nanocomposites lies in their ability to combine distinctive properties of both the matrix material at the nanoscale. This synergistic assimilation results in materials with improved strength, stability, conductivity, and other desirable characteristics. Moreover, the precise control over the composition, and morphology, allows the customization of nanocomposites for definite applications, revealing new possibilities for innovation and advancement. Harnessing the unique characteristics of nanocomposites offers unprecedented opportunities to address longstanding challenges across various fields, from materials science and engineering. At the heart of the fascination with nanocomposites lies their ability to seamlessly integrate the distinct properties of both matrix materials. This synergistic combination enables the tailoring of material properties with precision, unlocking countless functionalities unfeasible by conventional materials. Whether it's enhancing strength, improving conductivity, or conveying novel optical and electrical properties, nanocomposites offer a handy platform for innovation and advancement. Nanocomposites are composite materials whose smallest dimension is 100 nm or less. Nanocomposites have captured the interest of scientists all around the world with their distinctive design potential and multifunctional characteristics. The materials of the future are nanocomposites because of their versatility. Researchers from all around the world are developing a novel method for synthesizing nanocomposites to create materials with distinctive features. By combining proper constituents in proper proportion, nanocomposites can be synthesized, which gives better results in terms of efficacy, efficiency, *etc* [7-9]. Composite metal oxide nanoparticles have been extensively investigated for use in a variety of devices, including supercapacitors, sensors, solar cells, and photocatalytic systems, among others [10–14]. Further studies have shown that the properties of metal oxides can be altered by mixing them in proper proportion to increase their efficiency. This modification could lead to new applications in a variety of scientific fields, including chemistry, physics, biology, material science, and others [15]. Numerous nanocomposites made of metal oxides have been reported in the literature for different applications. The CuO-ZnO composite, which was reported as an antibacterial agent [16], and numerous more ones, like Graphene-ZnO used in photovoltaic cells [17], Polyaniline-ZnO nanocomposite sensors [18], and Graphene oxide/ZnO composite for photocatalytic degradation of dye [19], were described in the past few years.

This chapter aims to deliver a broad summary of the applications of nanocomposites in diverse fields, ranging from materials science and engineering to environmental

remediation. By delving into the particulars of nanocomposite design, fabrication techniques, and characterization methods, we aim to explain the underlying principles that govern their noteworthy properties and performance. The explicit objectives of this chapter are as follows:

- To discover the advanced techniques employed in the synthesis of nanocomposites, highlighting the importance of achieving a controlled growth process.
- To examine the mechanical, thermal, electrical, and optical properties of nanocomposites, clarifying the structure-property relationships that underpin their functionality and performance.
- To examine the applications of nanocomposites in materials science and engineering, including solar cells, gas sensors, supercapacitors, *etc.*

The present article briefly discusses the various synthesis methods for nanocomposite materials, mainly, hydrothermal, sol-gel, and wet chemical methods. This article also discusses some real-world uses in various metal oxide-based nanocomposites. As we navigate through these diverse applications, it becomes evident that nanocomposites represent more than just a technological advancement; they embody a paradigm shift in materials design and engineering. By harnessing the power of nanotechnology, researchers are not only unlocking new possibilities but also redefining the boundaries of what is possible, leading to a future where the intersection of science, engineering, and innovation knows no bounds.

## **SYNTHESIS TECHNIQUES OF MONCs**

Metal oxide-based nanocomposites, mainly, graphene-based composite metal oxides polymer-based nanocomposites are now the most widely used nanocomposites. The synthesis of nanomaterials has been regarded as the preferred subject in nanoscience since nanomaterials exhibit a variety of sized-based features. Researchers are working to develop new synthesis techniques that are quick, inexpensive, and environmentally benign, and whose results have great practical utility. The synthesis process for nanocomposites includes physical and chemical methods and transition metal oxides, which is summarised in this article. The methods that have been developed thus far include chemical vapor deposition, sol-gel methods, and hydrothermal methods. Now, we'll quickly go through each of these approaches.

# Highly Efficient Nanostructured TiO<sub>2</sub>@carbon Thin Film for Photocatalytic Degradation and Environmental Remediation: A Green Approach

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**Abstract:** The simple Chemical Bath Deposition (CBD) technique was used to create nanostructured TiO<sub>2</sub>@carbon thin films (TCTF) with improved photocatalytic properties. This research reports the modification of titanium dioxide using coconut husk fibre carbon. The first sol-gel method for the comparative low-temperature carbonization and acid digestion of coconut husk fibres has been suggested for the synthesis of carbon nanoparticles (CNPs) and their composite with TiO<sub>2</sub>. The microsphere-structured TiO<sub>2</sub>@carbon thin films were created by simply regulating the deposition process parameters. The morphology exhibits a strong correlation with the methyl orange (MO) photodegradation efficacy of TCTF as well. This discovery offers a suitable method for engineering the energetic and interfacial characteristics of TCTF to improve semiconductor photocatalytic performance. The anatase structure of the TCTF is visible in XRD. According to HR-TEM, TiO<sub>2</sub>@carbon nanocomposite (TCNCS) is prepared with a dimension of 10-15 nm. The Ti-O-Ti is strongly absorbed between 500 and 800 cm<sup>-1</sup> in both TiO<sub>2</sub> and the mixture, as shown by the FT-IR spectra. It can be seen from DRS spectra that the bandgap energy (E<sub>g</sub>) of TCNCS decreases significantly (3.05 eV). TCTF is composed of microspheres of various sizes and a smooth surface, according to FE-SEM images. Only Ti, C, and O are visible in the EDS result, demonstrating the great purity of the TCNCS made using this technique. Methyl orange (MO) degradation under UV light exposure was used to assess the photocatalytic activity of the TCNCS. The rate constant for TCNCS is greater than TiO<sub>2</sub>, and the photocatalytic degradation is observed to be pseudo-first-order.

**Keywords:** Coconut husk fibers, Cost-free, Carbon nanoparticles, Chemical bath deposition, Delay e<sup>-</sup>/h<sup>+</sup> recombination, Green nanomaterials, Increased degradation rate, Metal oxide, Methyl orange dye, Microsphere structures, Nanostructures, Nonmetal support, Photostable, Photocatalyst, Photocatalysis, Photodegradation, Semiconductor, TiO<sub>2</sub> nanoparticles, TiO<sub>2</sub>@carbon thin film, UV active.

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## INTRODUCTION

Major applications such as water splitting, CO<sub>2</sub> and N<sub>2</sub> fixation, as well as the decomposition of organic substances, and semiconducting photocatalysts have attracted a lot of attention [1-4]. Due to its higher photocatalytic activity, excellent photostability, non-toxicity, and low cost, TiO<sub>2</sub> is by far the most widely used [5]. However, the wide energy band gap of TiO<sub>2</sub> (3.2 eV) necessitates that only UV light with a wavelength range below 387 nm is used for excitation, which lowers the use efficiency of solar energy to a maximum of 6 % and prevents the commercialization of this technology. Therefore, the use of new visible-light-driven photocatalysts [6] has drawn a lot of focus in an effort to overcome the drawbacks.

The ability to postpone the quick recombination of photo-generated electron and hole pairs as well as the potential to absorb visible light are crucial for semiconductor photocatalysis to prove functional [7]. Amazing success was made, and several photocatalysts, primarily made of metal oxide semiconductors like TiO<sub>2</sub> [8], ZnO [9], ZrO<sub>2</sub> [10], SnO<sub>2</sub> [11], WO<sub>3</sub> [12], and CuO [13], have been used as effective photocatalysts. Due to its photostability, non-toxicity, and affordability, TiO<sub>2</sub> is one of these oxides that are frequently used as a photocatalyst.

Although TiO<sub>2</sub> is economical and environmentally favourable, its wide band gap necessitates the use of an e<sup>-</sup>/h<sup>+</sup> couple that allows UV light to start the photocatalytic process. Therefore, numerous alterations are made to TiO<sub>2</sub> to try and address these flaws and drawbacks, including the creation of hybrid composites, changing particle dimensions, the addition of dopants, depositing metal, and subsequent utilization of an optional element, *etc.* During this time, the frequency of the pair of electrons and holes exchange is slowed down in order to improve the photocatalytic activity. However these modifications have negative effects like photo-corrosion and restricted photo-stability. If the layer of TiO<sub>2</sub> was sustained by corrosion-resistant material, it is anticipated that TiO<sub>2</sub> would be an effective photocatalyst. Being an n-type, TiO<sub>2</sub> is with a spectrum of less than 380 nm for UV responsiveness.

Nanomaterials made from plants have made it possible to solve environmental problems in a novel, environmentally friendly way. It would be nice to make CNPs using simple, inexpensive, and environmentally sound techniques [14, 15]. Easy production of CNPs is possible, particularly with the aid of Coconut Husk Fibers (CHF). Already attempts have been made to generate CNPs by using coconut [16, 17]. The CHF's biomass waste is made up of cellulose (glucose), which is found

extensively in nature, hemicelluloses (pentose, hexose, and sugar acids), and lignin (aromatic alcohol). For the green synthesis of CNPs, this lignocellulosic biomass (LCBM) is the most prevalent, abundant, recyclable and natural material [18, 19]. As a result, it is now a potential source of carbon. A rare form of oxide semiconductor having good quantum efficiency, CNP is a brand-new, extremely effective form of carbon material [20, 21]. When compared to other support materials, its addition successfully increases the catalyst's specific surface area, boosts light absorption, and slows the rate at which photogenerated electron holes recombine. Key functions of CNPs in photocatalysis are: 1) On the exterior of CNPs, organic vibrant hues and pollutants are trapped, where they immediately oxidize and speed up photodegradation. 2) Because CNPs are black bodies, they act as superb carriers and boost a photocatalyst's ability to absorb light. 3) A plentiful and sustainable raw material, coconut husk fiber is used to make CNPs.

Numerous TiO<sub>2</sub>@carbon nanocomposite (TCNCS) photocatalysts have been developed as a result of flexible features and different kinds of carbon, including carbon nanotubes (CNTs) [22], graphene [23, 24], graphene oxide (GO) [25, 26], reduced graphene oxide (RGO) [27, 28], amorphous carbon [29, 30], carbon quantum dots [31, 32], graphitic carbon [33, 34], and C60 [35, 36]. Combining TiO<sub>2</sub> and carbon strengthens the overall impact of the photocatalytic reaction, particularly when exposed to visible light, and produces a synergistic reaction between the two carbon phases. This impact typically manifests as a decrease in the composite's band gap energy, which drives the photocatalytic reaction [37].

TiO<sub>2</sub>@carbon nanocomposite (TCNCs) with improved photocatalytic properties have recently been shown to be effective UV-visible light-driven photocatalysts. The subsequent study revealed that TCNCS was also effective at photocatalytic mineralization of both toluene [38] and CH<sub>3</sub>CHO [39]. The production of nanostructured TCNCs, specifically the nanoplates by physical vapor deposition (PVD) technique, Magnetron Sputtering [40, 41], and Chemical Bath Deposition (CBD) [42] showed excellent results. The TCNCs' production by calcining complex precursor [43] has lately been successful in increasing their photocatalytic activity. The hydrothermal method was also used [44] to produce the TCNCs. Particularly, TiO<sub>2</sub>@carbon thin films (TCTF) exhibit greater activity and may be used in solar-powered environmental cleanup [45]. TCNCs are unfortunately more difficult to dissolve in water. Since the photocatalytic reaction is a surface reaction, it is imperative to expose the surface of TCTF to contaminants and light in order to overcome the prior disadvantage.

## CHAPTER 5

# Bandgap Engineering and Optical Characterizations of Filler Reinforced PMMA Composite Thin Films

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**Abstract:** Polymeric composite (PC) materials are multifarious materials widely used in almost all industries due to their fascinating properties of being flexible, lightweight, durable, cost-effective, and easy mass fabrication in a variety of shapes and sizes. Furthermore, the thermo-physical properties of these polymeric materials can be further enhanced by the addition of an appropriate amount of organic or inorganic filler. Their high refractive index renders them to be used as components in the manufacturing of optoelectronic devices and hence certain optoelectronic parameters can be tailor-made by insertion of an appropriate filler in the host polymer. PMMA is one such versatile polymer with interesting optical properties, which can be further tuned up with filler enforcement for desired applications. This review deals with such organic and inorganic filler-doped PMMA composites with enhanced optical properties. Initially, the authors throw light on general physical and chemical properties of PMMA and its suitability to incorporate various fillers and the varied approaches of PMMA filler interactions. The review addresses briefly the various techniques of synthesis and optical characterisation of these PMMA-based PC. Further it attempts to summarize the underlying theories and concepts that construe the correlation between structure and optical parameters. The introduction of filler to bring a change in optical behaviour as desired is a challenging one. Hence authors have included not only the present state of art of these materials and the challenges thrown but also how the researchers are aiming to mitigate them in future.

**Keywords:** Absorption coefficient, Band gap energy, Extinction coefficient, Electrical resistivity, Finesse coefficient, High-frequency dielectric constant, Kramer's-Kronig's relation, Localized charge density value, Optical mobility, Oscillator strength, Optical conductivity, Organic and inorganic nanoparticles, Polymethyl methacrylate (PMMA), Polymer nanocomposites.

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## INTRODUCTION

Poly(methyl methacrylate) (PMMA)-based polymer nanocomposites have gained significant attention due to their unique thermos-physical properties and potential applications in various areas of optoelectronic devices, energy storage applications, electromagnetic shielding, *etc.* [1-12] The study of optical parameters of PMMA-based polymer nanocomposite thin films is of high significance in the industrial arena where PMMA nanocomposite films are used in various applications such as in coatings, as adhesives, and in lithography to organic light-emitting diodes and various organic material-based devices, including sensors and detectors [12-17]. Understanding the optical properties of these films is crucial for optimizing their performance in these devices.

Band gap engineering and optical characterization of filler-reinforced PMMA thin films involve the modification and study of the electronic band structure and optical properties of these nanocomposite materials. This is an essential aspect of tailoring the material for specific applications in areas like photonics, optoelectronics, and sensors. To achieve the desired optical properties in PMMA nanocomposite films, it is crucial to carefully select the type and concentration of nanoparticles, optimize dispersion techniques, and control film thickness. Characterization techniques such as UV-Vis spectroscopy, ellipsometry, and microscopy then help assess and analyze the optical properties of these materials. Additionally, the specific applications and requirements determine the most suitable design and formulation for PMMA nanocomposite films with tailored optical properties. Thus, to comprehend these aspects, the authors, in this chapter, review the variation obtained in various optical parameters such as energy gap, absorbance, absorption coefficient, extinction coefficient, finesse coefficient, refractive index, parts dielectric constant, and optical conductivity on incorporating various fillers in PMMA host matrix. The polymer-filler interaction is approached initially, then we laid down the theoretical considerations for the significant optical parameters. Here, we also discuss the reasons why PMMA is preferred as a host matrix. In the subsequent section, we have described the various methodologies like in situ polymerization, solution casting, and melt extrusion for the synthesis of nanostructured inorganic-organic PMMA films. This will present the advantages and disadvantages of these methods determined by the relation and route to the polymer-nanoparticle pair. This study is significant as this approach allows for tuning the absorption and emission characteristics of the films, leading to novel optical behaviors not achievable with pure PMMA films. The study aims to understand how the interaction between various fillers and host polymer matrix takes place to improve the optical parameters and manipulate band-gap to create



materials with novel properties to be used in areas such as optoelectronics, photovoltaics, sensors, and displays. Further section will contain a discussion on the various techniques of measurement of optical parameters. Finally, the current state of the art of these materials and their potential applications; the challenges, and the scope of future development shall be referred to.

### **Approaches to Polymer Filler Interaction**

When the filler is dispersed in the polymer matrix, the surface area of the filler will form an “interaction zone,” leading to changes in polymer behaviour, morphology, space charge distribution, and bond dispersion [18-19]. The interaction between the filler and the polymeric matrix in nanocomposites is a complex phenomenon that depends on various factors such as the nature of the filler, its surface chemistry, and the compatibility between the filler and the polymer matrix. The interaction can be influenced by parameters such as particle size, shape, surface area, and surface functionalization [18]. The interaction between the polymer and the filler in nanocomposites can alter the optical properties of the material. This can be attributed to the interaction between the fillers and the polymer matrix, which affects the electronic structure of the material [17-18]. The refractive index of PMMA nanocomposites can be influenced by the presence of fillers. Changes in the refractive index can affect the optical properties, such as light propagation and reflection, of the material. The density of the filler determines how strongly the light is scattered within the material. The low filler proportion regions show lower scattering than denser filler regions [19]. The addition of fillers can alter the absorption and transmission properties of PMMA nanocomposites.

When fillers are added to a PMMA host matrix, the band gap of the composite can change. This change in band gap is primarily attributed to the interaction between the fillers and the host matrix, as well as the inherent electronic properties of the fillers themselves. The key factors explaining why the band gap of PMMA changes upon filler incorporation:

*Refractive Index Matching:* Fillers often have different refractive indices than PMMA. When the refractive indices of the filler and the matrix do not match, there can be significant reflection and refraction at the filler-matrix interfaces. This can lead to increased light scattering and reduced transparency, affecting the overall optical clarity of the composite [19-20].

*Quantum Confinement:* In the case of nanoscale fillers quantum dots are added to the PMMA matrix, so quantum confinement effects can occur. These effects arise

## CHAPTER 6

# Electrodeposited Bismuth Oxide Electrodes for Energy Storage Applications

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**Abstract:** The present topic is focused on the synthesis of bismuth oxide thin films on different substrates using the electrodeposition technique. Prepared samples were annealed at 573 K and further used for physical and electrochemical characterizations. The structural investigations of the prepared samples show polycrystalline nature with a tetragonal crystal structure. Morphological analysis shows spruce leaves with nano-rod-type architecture morphology. All samples show hydrophilic nature. Specimens for electrochemical analysis were scanned by CV in 1 M KOH on numerous scan rates, starting from 1 to 100 mV/s. The obtained maximum value of specific capacitance (SC) is 1742.79 F/gm. at a scan rate of 2 mV/s in 1 M KOH with a potential window (– 1.0 V to 0.8 V) vs. Ag/AgCl and acquired maximum value of specific energy and specific power was 56.68 Wh/Kg and 2.94 kW/kg at 10 mA/cm<sup>2</sup>. The Nyquist plot shows the internal resistance of the optimized electrode. The final optimized sample will be used for the energy storage supercapacitor application.

**Keywords:** Bismuth Oxide, Bi<sub>2</sub>CuO<sub>4</sub>, Charge-discharge, Cyclic voltammetry, Electrodeposition, Electrochemical characterization, Energy, Electrolyte, EDAX, Electrode, Impedance, Mapping, Power, Stability, Substrates, SEM, Specific capacitance, Supercapacitor, Thin film, XRD.

## INTRODUCTION

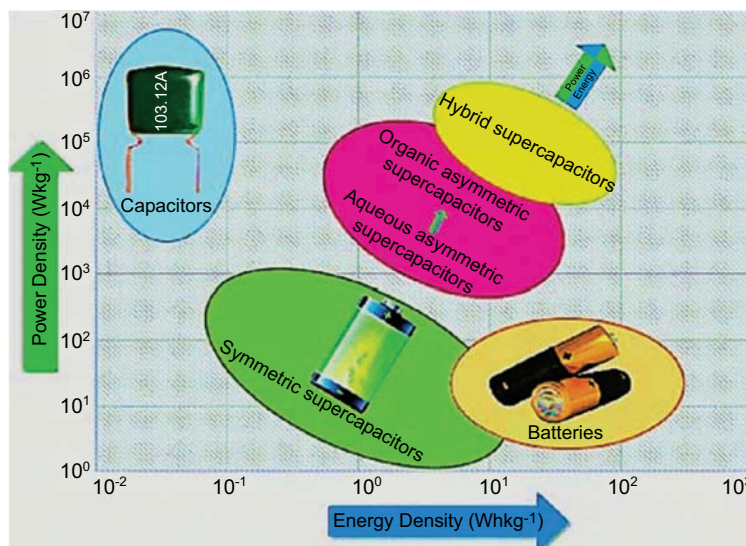
To accommodate the rising need for electricity and power in our everyday lives, it is essential in the modern world to utilize energy storage systems with high power density, extensive life cycles, and quick charging [1]. This is a result of the quick-growing and constant need for versatile and transportable advanced technologies. Because of the lack of supply of energy, for portable electrical devices that demand

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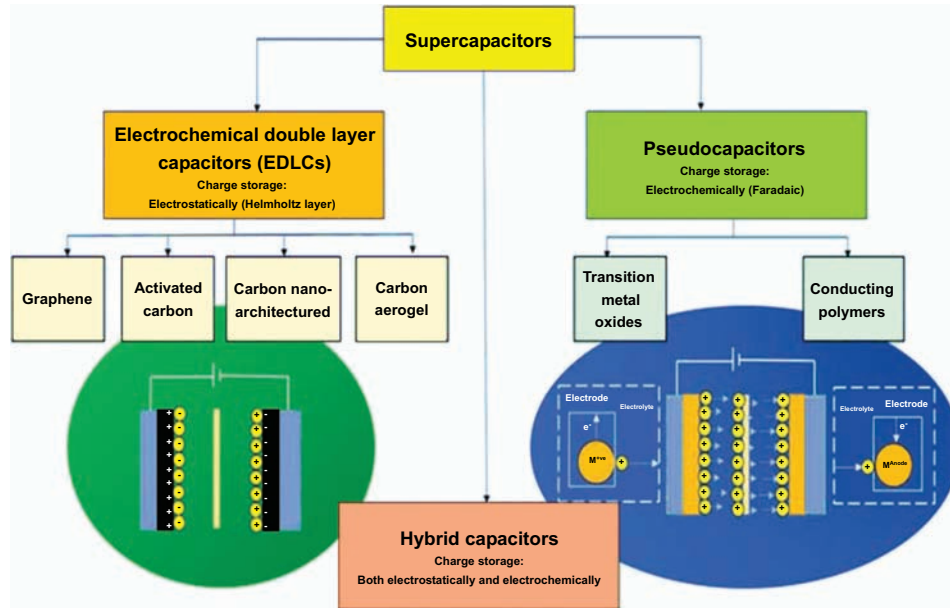
enormous energetic density and power densities, ordinary capacitors and batteries are adequate. These will lead to an enlargement of brand-new kinds of energy-storing devices designated as a supercapacitor [2]. Fig. (1) shows the Ragone chart for several energy storage devices, including fuel cells, standard batteries, supercapacitors, and traditional capacitors. The specific energy SE (Wh/kg) and specific power SP (W/kg) for the device are depicted in this graph as a function of discharge time. Batteries have an extraordinary energy density (100–200 Wh/kg) but their SP performance degrades because of their prolonged recharging times. Due to their higher SP (500–10,000 W/kg) relative to batteries and superior SE (1–10 Wh/kg) relative to conventional capacitors, supercapacitors are recognized as an essential part of the energy storage class. Supercapacitors can be completely charged and drained at an appropriate quick pace, and they have a longer cycle lifespan. With a significant power density and even a prolonged life cycle, SCs have become prominent as prospective energy storage technologies. To increase the functionality of SCs, scientists are now concentrating on the manufacturing of highly electroactive substances, which is a crucial hurdle to overcome.



**Fig. (1).** A graph illustrating specific energy against specific power for several kinds of energy storage systems [3].

Depending on the charge-storage properties of the electrode materials, specific capacitors are mostly categorised as EDLC capacitors, pseudocapacitors, and hybrid supercapacitors [4]. Classifications of supercapacitors are depicted in Fig. (2). For maintaining the charge that characterizes it, each class has a special way. These fall into three categories: faradic, non-Faradic, and a hybrid combination of

the two. The exchange of charge between an electrode and an electrolyte is a crucial component of faradic processes like oxidation-reduction reactions. Instead of relying on a chemical reaction, a non-Faradaic process does not. Charges on surfaces are instead distributed through physical processes rather than ones that involve the formation or severing of chemical connections.



**Fig. (2).** Types of supercapacitors [5].

### EDLC Supercapacitors

Significant interactions among the ions and molecules in the solvent and electrode surface lead to the formation of an electrical double layer. A thin layer of charge forms on the metal surface when there is a deficiency or deficit of electrons at the metal contact. However, due to the numerous cations or anions present there, the solution at the electrode surface has the opposite charge. All the charged entities and oriented dipoles that exist at the metal-solution interface are consequently comprised of an electrical double layer. The double-layer concept is used to store energy in the EDLC. This double layer is produced when two electrodes dipped in an electrolyte are polarised, as shown in Fig. (3). Conventional capacitors have limited ability to store charges and structural restrictions on the space among the dual-charged plates prevent them from storing much energy. The enormous interfacial surface as well as the atomic array of charge segregation lengths in supercapacitors depending on the EDLC method, allow for far more energy storage.

## Metal Oxide Thin Films: A Comprehensive Study of Synthesis, Characterization and Applications

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**Abstract:** Nanometer-accurate surface coverage has become achievable through improvements in thin film deposition methods, enabling scientists to construct multilayers with complex compositions and investigate the cumulative effects of their interactions. Furthermore, enhancements to the deposition procedure have made it possible to produce significantly smaller electrical devices, which is crucial for introducing cutting-edge technology. The development of nanotechnologies, such as thin films, requires stringent control over the deposition process to minimize the physical dimensions of devices during manufacturing. Continued research in this area can benefit photovoltaic devices with anti-corrosion or biocidal coatings to meet the requirements of contemporary society. This chapter discusses the relevance of metal oxide thin films and various manufacturing methods. We also review different characterization techniques, including electron microscopy, x-ray diffraction, atomic force microscopy, Fourier transform infrared spectroscopy, photoluminescence, and UV-visible spectroscopy. We emphasize the various applications of these metal oxide thin films.

**Keywords:** Applications, Chromic devices, Fourier transform infrared spectroscopy, Gas sensors, Metal oxide, Scanning electron microscopy, Thin film characterization, X-ray diffraction, X-ray photoelectron spectroscopy.

### INTRODUCTION

Recently, nanomaterials comprised of metal oxides have generated considerable scientific and technological interest and innovation. Because of their exceptional

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physical, chemical, and optical characteristics, they have various practical applications [1]. The deposition processes that can be employed to achieve precision control over the thickness of these materials span from a few nanometers to several micrometers [2,3]. These processes include chemical bath synthesis, physical or chemical vapor deposition, sputtering, and atomic layer deposition. The exceptionally high surface area to volume ratio of thin-film nanomaterials sets them apart. This property improves the electrical and optical properties of the substance, in addition to increasing its reactivity and catalytic activity. Because of their multifunctional qualities and distinctive characteristics, these materials are in great demand for an extensive range of applications [4–6].

Thin-film metal oxides, which are transparent and conductive, find widespread application across various technological fields. For instance, their capacity to conduct electricity and permit light transmission is utilized to optimize the efficiency of energy conversion in solar cells, where they function as electrodes [7]. Furthermore, their outstanding chemical stability, gas-reactive characteristics, and expansive surface area render them highly suitable for gas-sensing applications [8]. Therefore, their sensitivity and selectivity are significantly enhanced. Thin-film metal oxides have been used as catalysts in industrial processes and to clean up the environment, and this has mostly worked because of their unique surface properties [9]. Because of their exceptional capacity and extended lifespan, these substances are exceptionally well-suited for implementation as electrode materials in energy storage apparatus, including batteries and supercapacitors [10]. The ability to precisely regulate the deposition process of extremely thin metal oxide coatings facilitates the manipulation of light absorption, transmission, and reflection. Subsequently, substantial progress has been made in the domains of optics and photonics [11].

On the contrary, their remarkable electrical characteristics render them highly compatible with operating as active layers in electronic devices, thereby propelling progress in fields like semiconductor technology [12]. Because of their biocompatibility and ability to interact with biological systems, thin-film metal oxides have drawn increasing interest from the biomedical community. Among other applications, they are utilized in scaffolds for tissue engineering, drug delivery, and biosensors [13].

The main objective of this chapter is to present a thorough overview of the fabrication process, characteristics, and pragmatic implementations of metal oxide thin-film nanomaterials. These materials have attracted growing attention due to their extensive potential applications. The principal topics of discourse will

comprise nickel oxide, zinc, titania, alumina, and copper. In this investigation, we shall examine various deposition techniques employed in fabricating these thin films, assess their unique characteristics, and examine their multifarious implementations in various scientific and technological fields. Through our analysis, we aim to shed light on the present comprehension of metal oxide thin film nanomaterials and inspire further developments in this swiftly progressing study area.

This chapter aims to thoroughly explain metal oxide thin film nanomaterials, including a clear description of their properties, techniques for synthesizing them, and their potential applications. While our chapter provides a unique viewpoint by consolidating data from several sources and presenting it as organized and understandable, there is existing academic research on subjects relevant to metal oxide thin films. Our study focuses on the benefits of the deposition processes used to prepare the metal oxide thin films. The study also encompasses several scientific and technological uses of metal oxide thin films, such as copper oxide, nickel, zinc, titania, and alumina. Our chapter compiles information on these materials, making it an excellent resource for academics, engineers, and practitioners interested in metal oxide thin films. The aim is to help individuals unlock their creative capacity for practical use.

## **SYNTHESIS OF THIN FILM METAL OXIDES**

Various techniques and substrate materials can produce different types of thin films. The method and substrate material used for a process depend on the size of the layer required, the application of the thin film, and the number of layers needed for the process. Some techniques that various researchers commonly use to produce thin films are sputtering atomic layer deposition and the sol-gel method.

In the study, Mustaffa *et al.* [8] utilized radio frequency (RF) magnetron sputtering to generate ZnO thin films. Glass substrates were subjected to high-temperature sterilization and cleaned with acetone and distilled water before deposition. The substrates were then inserted into the magnetron sputtering system, and a spacing of 21 cm was set between the target cathode and the stainless-steel substrate holder. The pressure inside the sputtering chamber was lowered to 50 mTorr using turbomolecular pumps, and 150 W of radiofrequency radiation was used to make plasma. The oxygen-to-argon ratio was regulated using a 4% mass flow controller. The samples were deposited by sputtering for three hours at room temperature using a circular zinc target.

## CHAPTER 8

# TiO<sub>2</sub>, ZnO and Fe<sub>2</sub>O<sub>3</sub> Thin Film Nanomaterials: Preparation to Applications

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**Abstract:** Nanomaterials have gained a lot of attention of scientists and researchers during the last two decades due to their small size (nano-scale) and large surface area. Amongst these nanomaterials, metal oxide thin film nanoparticles are gaining much more interest due to their exceptional chemical, electronic, catalytic, electrical and optical properties. These properties can be improved to develop essential functionalities and compositions that make them fit for various applications such as catalysts, solar cells, sensors, optoelectronic materials, and green energy storage applications. Thin film metal oxide nanoparticles can be synthesized by different physical or chemical methods like physical vapour deposition, chemical vapour deposition, atomic layer deposition, sol-gel synthesis and hydrothermal synthesis. The usual characterization techniques for metal oxide nanoparticles are SEM, HRTEM, EDX analysis, XRD, FTIR, XPS, and TGA–DTA *etc.* Many metal oxides like TiO<sub>2</sub> and ZnO have excellent properties like photo-induced phenomenon under UV radiation and superconducting properties. Thus, their thin film nanoparticles can work more efficiently than the bulk one. This chapter explains about the synthesis of some metal oxides like TiO<sub>2</sub>, ZnO, and Fe<sub>2</sub>O<sub>3</sub> through various physical and chemical methods, and the characterization and application of metal oxide thin film nanoparticles for solar cells, fuel cells, photovoltaic cells, optoelectronic application, and green energy storage application.

**Keywords:** Iron oxide, Li ion battery, Nanomaterials, Supercapacitor, Solar cell, Titania, Zinc oxide.

## INTRODUCTION

In the recent past, nanotechnology has drawn a lot of attention from the scientific community. Here, nanoparticles (NPs) are used for various purposes viz. photocatalysis, catalysis, electrochemical sensing of pollutants, drug delivery, solar cells, *etc* [1]. Richard P. Feynman, a physics Nobel laureate, was the first one to introduce the concept of nanotechnology at the meeting of the American Physical Society in 1959 in his lecture titled “There’s Plenty of Room at the Bottom” [2].

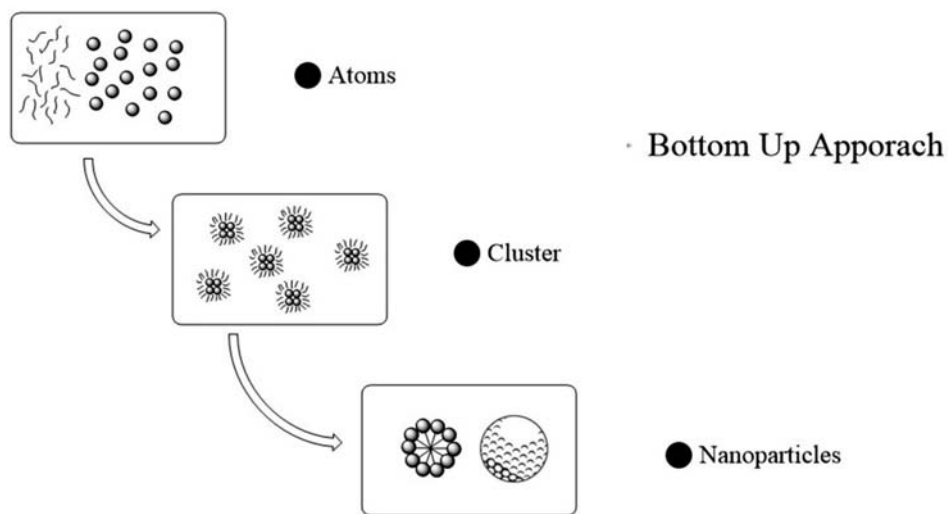
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The significance of nanomaterials in the arena of science and technology relies on their synthesis methods and modulation, which result in considerable modification in the properties of nanoparticles [3]. For synthesizing nanomaterials, two approaches are used the ‘top-down approach’ including physical and chemical methods, and the ‘Bottom-up approach’ including biological and chemical methods [4].

The bottom-up approach depends on the fact that how appropriate “metal-organic” molecules are being selected as precursors, here atoms or molecules combine on their own and get converted into nanosized structure. The ‘Bottom-up approach’ is considered better than the ‘Top-down approach’ as it is easy to control the particle size and shape of the synthesized nanomaterials [5]. Hydrothermal, solvothermal, precipitation, sol-gel method, electro-chemical deposition, spray pyrolysis, *etc.* are a few examples of the ‘Bottom-up approach’ [6]. This is a kind of self-assembly of atoms or molecules. Self-assembly means the attachment of molecules in a particular manner on or to particular surfaces, which is basically related to the self-ordering principles. We can understand it from the example of DNA where a particular DNA strands is attached to its complementary base pairs. In a similar fashion, in ‘Bottom-up approaches’ molecular building blocks behave as if they are a specific part of a jigsaw puzzle and they assemble in a particular manner without any driving force to make a meaningful unit (Fig. 1) [7].



**Fig. (1).** Schematic Representation of Bottom-Up Approach.

At present, transition metal oxide nanoparticles (TMONPs) are of great significance and have potential market value due to their interesting properties and significant

application in various fields when they are brought to the nanometre range. Interest in the TMONPs lies in their wide number of applications ranging from catalysis to drug delivery to environmental remediation [8]. For the application of these TMONPs in various fields, it is desirable to synthesize them in a controlled manner giving precise shape, size and band gap energy, which is easy to achieve by solution-phase methods. By engineering the required TMONPs, we can also rule out the limitations faced during the use of their bulk counterpart, which helps in making amazing breakthroughs in various arenas [9].

Zinc oxide (ZnO) in nano regime is nontoxic and biocompatible. Many of its unique properties arise from its size, shape, and morphology [10]. ZnO NPs are used in personal care products, such as cosmetics, ointments, and sunscreen as they exhibit strong UV absorption properties. They are also used in the rubber industry as their composites give rise to a wear-proof rubber with improved performance, toughness, intensity, and antiaging properties [11,12]. When Zinc Oxide NPs are mixed with the fabrics, they demonstrate attractive functions like resistance to ultraviolet and visible light and are unaffected by bacteria and fungi. Food and Drug Administration (FDA) has also nodded for the use of Zinc Oxide NPs in a novel and effective antitumor therapy as they have innate anticancer properties [13].

TiO<sub>2</sub> NPs find great application in industry and consumer products as they exhibit strong catalytic activity, which is ascribed to their smaller sizes, resulting in a high surface-to-volume ratio. TiO<sub>2</sub> is found mainly in three phases- anatase, rutile, and brookite titania. Out of three, anatase titania has received considerable attention due to self-cleaning properties (the capability of degrading air pollutants *via* photocatalysis) in the construction industry [14]. On mixing with cement, these NPs neutralize organic pollutants, which are removed *via* the hydrophilic character of the plane without affecting the features of concrete structures, especially in the case of white cement [15].

The chemical combination of iron and oxygen generates iron oxides, and ~16 iron oxides are identified. We can see, iron (III) oxide (Fe<sub>2</sub>O<sub>3</sub>) in the form of rust naturally [16]. Magnetite (Fe<sub>3</sub>O<sub>4</sub>), Maghemite ( $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>), and Hematite ( $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>) are the three major forms of iron oxide. Iron oxides are used extensively as they are economic, and also have a vital role in many biological and geological processes. Besides, iron oxides find applications in catalysis, and durable pigmentation (coating, painting *etc.*) [17]. Iron oxide NPs exhibit super-para magnetism at sizes below 10-20 nm. Fe<sub>3</sub>O<sub>4</sub> and  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> show low toxicity, high surface-to-volume ratio, and simple separation methodology so find great applications in the

## CHAPTER 9

## Thin Film of Molybdenum Disulphide, an Emerging Class of Semiconducting Materials: Properties, Characterization and Photovoltaic Applications

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**Abstract:** Several scholars and scientists have recently continued their efforts to fabricate and develop advanced nanomaterials in the form of nanoparticles, clusters, emulsions, and thin films to design nanoscopic optoelectronic devices, supercapacitors, solar systems, and biomedical equipment. Because of the widespread exceptional physicochemical characteristics and improved functionalities, hybrid nanostructures, including organic and inorganic metal-oxides, sulphides and polymeric nanostructures are highly appreciated and explored for enhanced physicochemical, biological, and environmental applications. Therefore, metal-sulphides nanomaterials such as CdS, ZnS, MoS<sub>2</sub>, and PbS, as nano-thin films were widely designed, and employed in various geometries such as 1D, 2D, and 3D nano-thin films, which possess extraordinary functionality. Among them, MoS<sub>2</sub> (molybdenum disulfide) is considered as an emerging class of semiconducting material due to its direct bandgap value *i.e.* (~1.9 eV), has high current on/off ratio (10<sup>8</sup>) at normal temperature, and exhibited mobility 200 cm<sup>2</sup> Vs<sup>-1</sup>. It has the ability to change its architecture from bulk to nanoscale level. On the basis of its unique structure, MoS<sub>2</sub> has two characteristics: (i) it possesses a hexagonal structure with S-Mo-S layers arrangement by covalent bond, and (ii) Van der Waals force of interaction that lies between the adjacent layers of MoS<sub>2</sub>, which makes it suitable for multiple applications. Moreover, the structural, surface, and optical properties of MoS<sub>2</sub> are altered by the stoichiometric doping of metal/ions, which favour its electronic features toward improved work functionalities. This chapter will provide a systematic explanation for the synthesis, design, morphological investigations, and developments of the MoS<sub>2</sub> semiconducting nano-thin films for multiple optoelectronic, biochemical, and environmental uses.

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**Keywords:** 2D nano-thin films, MoS<sub>2</sub>, Modified structures and surfaces, Solar cells, Sensor applications.

## INTRODUCTION

Recently, there has been a significant interest in the synthesis, characterization, and application of semiconductor nanoparticles, which have emerged as a key component in many new technologies [1-3]. When semiconductor materials are reduced to the nanoscale, their physical and chemical properties undergo significant changes, resulting in unique properties that are attributed to their large surface area and quantum size effects. This alteration in their properties can affect their conductivity, as well as their optical properties such as absorption coefficient and refractive index. Although semiconductor nanomaterials and devices are still in the research phase, they hold great promise for a wide range of applications in various fields, such as solar cells, electronic devices at the nanoscale, light-emitting diodes, laser technology, and catalysts [4-6]. Nanotechnology advancements are expected to bring about significant breakthroughs in the semiconductor industry, enabling the development of various diodes, such as light-emitting diodes, silicon-controlled rectifiers, and digital and analog integrated circuits. Several semiconductor nanomaterials, including Si, Si-Ge, GaAs, AlGaAs, InP, InGaAs, GaN, AlGaN, SiC, ZnS, ZnSe, AlInGaP, CdSe, CdS, and HgCdTe have found extensive applications in numerous electronic devices [7-9]. The chapter aims to provide an overview of the synthetic methods and applications of semiconductor nanoparticles, specifically focusing on Molybdenum disulphide semiconductor nanoparticles, which exhibit quantum confinement behavior within the 1-20 nm size range [10, 11]. Thin films have been utilized for over 50 years in the production of electronic devices, decorative parts, optical coatings, and hard instrument coatings. The processing of thin films is a well-established technology for material processing, where the deposition of material atoms on a substrate is used to create a low-dimensional material, one atomic/molecular/ionic species at a time. Typically, the thickness of a thin film is less than a few micrometers, making its dimensions comparable to those of nanomaterials. Thin-film technology is widely employed for processing nanomaterials, and therefore, thin-film materials fall under the same category as nanomaterials [12-14].

Two-dimensional materials, specifically transition metal disulfides (MS<sub>2</sub>), such as MoS<sub>2</sub>, TiS<sub>2</sub>, WS<sub>2</sub>, and VS<sub>2</sub> are highly sought after for use in future nanoelectronic devices due to their unique properties [15, 16]. These inorganic layered compounds have been found to have potential applications in various fields, including catalysis, transistors, hydrogen storage, solid lubricants, Li-ion batteries, and double-layer

capacitors. Moreover, the thin films of metal chalcogenide material have gained tremendous attention in the research field. The deposition of thin and fine continuous layers of materials over other base materials, which can be metal, ceramic, *etc.* by using so many physical, chemical, mechanical, and electrical methods. The fabrication of thin film by the above method is so easy and this process is called thin film deposition. The energy generation by thin film deposition technique is very useful in the semiconducting field because the surface properties, including reflection, transmission, absorption, stability, corrosion resistance, permeation, and electrical behavior of materials are improved. Moreover, the thickness of the thin film is easily controllable as per requirement. Owing to the above excellent properties of thin film, the MoS<sub>2</sub> thin film has gained tremendous attention in the research field. The sheet-like morphology of MoS<sub>2</sub> has shown promising applications in the semiconducting field. Bulk MoS<sub>2</sub>, for example, is an indirect semiconductor with a bandgap of 1.2 eV. The inorganic compound MoS<sub>2</sub>, a member of the transition metal dichalcogenides (TMDs) series, consists of one Molybdenum atom and two Sulphur atoms. Monolayer MoS<sub>2</sub> is a thin, transparent semiconductor with a direct bandgap of 1.8 eV, making it a potential candidate for use in optoelectronics and energy harvesting applications. This material could work in conjunction with graphene to fulfill such requirements. Whereas the sandwich sheets interact *via* comparatively modest van der Waals forces, covalent bonding in a succession of S-Mo-S. Every sheet typically has a thickness of 0.65 nm. It has been discovered that monolayer MoS<sub>2</sub> with trigonal prismatic polytype is semiconducting (2H), but monolayer MoS<sub>2</sub> with octahedral structural geometry arrangement is metallic (1T), including 3R (rhombohedral) [17]. As a substitute for graphite in high-vacuum operations, MoS<sub>2</sub> is also used, but its maximum operating temperature is lower than that of graphite. Bulk MoS<sub>2</sub> is a semiconductor with an indirect bandgap of about 1.2 eV, and as such, is only of limited importance to the nanoelectronics sector [18, 19].

This chapter serves as a comprehensive resource, offering fundamental insights into the formation of MoS<sub>2</sub> thin films through diverse techniques. It delves into the structural, optical, and surface characteristics of these films, synthesizing key research findings. The discussion extends to both pure MoS<sub>2</sub> structures and modified counterparts, elucidating their roles in enhancing solar cell performance while also presenting future perspectives. Furthermore, the chapter addresses novel strategies aimed at improving optoelectronic properties through innovative synthesis techniques. The overarching aim of this chapter is to provide valuable knowledge to academic and scientific scholars, facilitating a broader understanding of 2D nano thin film materials tailored for applications in energy storage and conversion systems. By elucidating the current state of research and offering

## Aligned Carbon Nanotube Thin Film Based High Energy Cathode Emitters

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**Abstract:** This chapter emphasizes on carbon nanotube's (CNT) growth kinetics and the development of CNT-based thin film cathode emitters. It highlights intensifying critical process parameters to design appropriate cathodes by creating vertically aligned carbon nanotubes using injection chemical vapour deposition. In the process of improving field emission (FE) characteristics, (i) the process of hetero-atom doping, (ii) high degree of alignment, (iii) controlled spatial distribution, and (iv) uniform height of CNT are the desirable criteria. The strategy of hetero-atom doping in a carbon network is adapted to tailor the work function of CNT, which is crucial in tuning the FE characteristics of CNT film. Doping-induced atomistic-defects influence field emission characteristics, while thermal transport and failure of carbon nanotubes with fast transient joule heating during the field emission are described with the experimental evidences. The electrostatic screening effects (ESE) between aligned CNTs and the strategies to suppress ESE were critically emphasized. In this chapter, the role of (i) island-structured CNT film, (ii) patterned growth, and (iii) height-to-diameter ratio modulation in suppressing ESE is elaborately discussed. The physical parameters of CNT and phenomenon affecting the electron emission characteristics of CNT cathode are described in detail. Ultimately, CNTs standing vertically on the substrates are noble candidates for constructing new cathode electrodes fulfilling these exceptional FE characteristics, but their dimensions and density on the substrate must be adjusted and tailored for real-time applications. Considering the importance of this application, the challenges and future prospectus of CNT based cathode emitters are also covered.

**Keywords:** Carbon nanotubes, Doping, Electrostatic screening, Field emission, Pattern growth, Thin film.

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## INTRODUCTION

The emission of electrons from a solid material is a surface phenomenon. It can be mainly categorised into (i) thermionic electron emission (TE), (ii) field-enhanced thermionic emission (FTE) and (iii) cold-field electron emission (FE). In any case (for TE, FTE, FE), the work function of the electrode plays a pivotal role in liberating the electron from its surface [1, 2]. TE is the liberation of electrons from the surface of the electrode by virtue of external temperature provided in the form of heating. In TE, the external thermal energy is applied to the thermions (electrons or ions), which aids them in overcoming the material's work function. The classical example of TE is the Edison effect, where the cathode (hot) emits electrons into a vacuum tube. The emission of electrons from the hot filament or metal occurs at elevated temperatures over 1000 K. The magnitude of the emitted electrons and charge flow increases steeply rise with a rise in temperature. The current from a heated wire appears to be exponentially dependent on the external temperature of the wire with an empirical relation similar to the Arrhenius equation. Lately, field enhanced thermionic emission is also called Schottky emission. The FTE is dominant for an electric field having strengths lower than about  $10^8$  V/m. With electric fields exhibiting strengths higher than  $10^8$  V/m, Fowler-Nordheim (FN) tunnelling plays a significant role in affecting the emission current. At uniformly high electric fields, FN tunnelling happens to be the predominant electron emission mechanism making the emitter operate in the cold-field electron emission [1,2]. For the emission of electrons from the electrode surface, TE, FTE and FE need a hot cathode, field-induced cathode and cold cathode, respectively. The extent of temperature and electric field involved in the emission of electrons is shown in Table 1. The details on the mechanism and material requirement for FE are explained in Section 2.1.

**Table 1. Comparison of emission mode depending on temperature and electric field.**

Emission mode	Temperature	Electric field
TE	High	Weak
FTE (Schottky)	Medium	Medium
FE	Low	Strong

The class of tubular-structured nanocarbon, called carbon nanotubes (CNT) have occurred as probable field emitters pertaining to low threshold electric fields and very high emission current density. The sharp tips, high aspect ratio, high electrical conductivity, elongated tubular structure, and concentric multi-shelled cylinders allow electron emission with low work function and fast tunnelling, which makes them highly attractive for technological applications in various kinds of vacuum-based electronic devices such as flat panel display units, microwave tubes, electron gun, lighting tubes, amplifiers for space communication, X-ray sources, *etc.* [3-11]. As compared to several thermionic emitters (conventional electron field emitters), such field emission-based cathodes operate at a relatively low applied voltage [1, 2], which could lead to the development of miniaturized compact vacuum appliances for a plethora of applications.

Also, the various parameters involved in influencing the FE characteristics of the CNT include: (i) aberration in the vertical orientation of nanotubes, (ii) optimum inter-tube distance in the CNT array, (iii) the electric field amplification factor near a CNT tip with dependence on the shape of the tip, (iv) spatial spread of each individual CNT comprising the cathode, (v) electric field screening by neighbouring CNTs, (vi) CNT-emitter deterioration mechanism due to constant ion bombardment of the tips due to electron impact ionization of the residual gaseous molecules, and (vii) thermal events causing the degradation of CNTs during emission. To derive at maximum reachable emission current of CNT arrays, the aforementioned factors are considered and optimized. Deriving CNTs that possess all the aforementioned characteristics is desperately desirable to achieve a high-energy cathode emitter. Moreover, the aligning of the CNTs in a particular direction to augment emission current is the peculiarity of such vertically-oriented CNTs perpendicular to the substrate.

This chapter mainly discusses experimental and theoretical methods and approaches applied for designing a cold emitter; especially, the growth kinetics of aligned carbon nanotubes to make a thin film and its impact on FE characteristics. The strategy of augmentation of emission current by realising the aligned carbon nanotubes and creating the island-structured CNT arrays from them with minimal screening effect is the prime objective of the book chapter.



## Exploring Quaternary $\text{Ni}_{0.70-x}\text{Cu}_x\text{Zn}_{0.30}\text{Fe}_2\text{O}_4$ Ferrimagnetic Thin Films for Gas Sensing

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**Abstract:** A nanocrystalline quaternary  $\text{Ni}_{0.70-x}\text{Cu}_x\text{Zn}_{0.30}\text{Fe}_2\text{O}_4$  ( $x = 0.00, 0.05, 0.15, \text{ and } 0.25$ ) ferrimagnetic thin film was deposited and studied utilizing advanced characterization techniques, including XRD, Raman spectroscopy, FESEM, AFM, XPS, *etc.* The details of the investigations made by these techniques into the structure, chemical environment, morphology, physical properties, and sensing are presented in this chapter. Spray pyrolysis was used to deposit quaternary ferrimagnetic thin films using metal nitrates as the starting material. The cubic structure was revealed by XRD patterns. The peaks in the Raman spectra correspond to the tetrahedral and octahedral sites supporting the formation of the cubic phase. The presence of permitted compositional constituents in the XPS indicates phase-pure production. FESEM images revealed some spherical agglomerations. The elemental composition was identified by the presence of Ni, Cu, Zn, Fe, and O elements in the EDS pattern. The FESEM cross section showed the deposition on the substrate, which is uniform and dense. The spherical shape, crack-free, and defect-free structure of deposited thin film system was observed by AFM. Contact angle measurements showed the thin films were hydrophilic. The sensitivity among  $\text{H}_2\text{S}$ ,  $\text{NO}_2$  and  $\text{NH}_3$  gases was shown by  $\text{H}_2\text{S}$  gas at an operating temperature of  $200^\circ\text{C}$  for a composition of  $\text{Ni}_{0.65}\text{Cu}_{0.05}\text{Zn}_{0.30}\text{Fe}_2\text{O}_4$ . The minimum detectable concentration was 20 ppm. With an increase in  $\text{H}_2\text{S}$  concentration, a linear improvement in the sensing response was seen. Additionally, it was discovered that the response time shrank with an increase in  $\text{H}_2\text{S}$  concentration. At 50 ppm  $\text{H}_2\text{S}$ ,  $\text{Ni}_{0.65}\text{Cu}_{0.05}\text{Zn}_{0.30}\text{Fe}_2\text{O}_4$  has shown extremely high repeatability.

**Keywords:** Cubic structure, Gas sensing, Raman spectra, Spray pyrolysis, Thin films, XRD, XPS.

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## INTRODUCTION

The rise of industrialization has led to an escalation in air pollution, driven by the evolution of pollutants. Concurrently, the global market for gas sensors, valued at USD 1.7 million in 2012, is projected to grow at a steady rate of 5.1% annually [1]. This underscores the importance of accurately measuring gaseous component concentrations. Chemical sensors play a crucial role in detecting poisonous, combustible, and flammable gases such as H<sub>2</sub>S, CO, CO<sub>2</sub>, NH<sub>3</sub>, Cl<sub>2</sub>, SO<sub>2</sub>, NH<sub>3</sub>, H<sub>2</sub>, CH<sub>4</sub>, LPG, ethanol [2-8], *etc.* Among these sensors, ferrimagnetic semiconductors stand out for their exceptional sensitivity, simplicity, selectivity, low maintenance, stability, rapid response, and cost-effectiveness. Their sensitivity stems from the oxidation or reduction of multivalent cations due to changes in oxidation states [9].

Hydrogen sulphide (H<sub>2</sub>S), a highly toxic gas, primarily originates from the decomposition of organic compounds [10-12]. Its presence in harsh environments necessitates specialized sensors capable of withstanding such conditions while delivering reliable signals. H<sub>2</sub>S not only poses health hazards but also induces pipeline corrosion, environmental contamination, and catalyst deactivation, leading to efficiency losses and structural failures. Developing efficient H<sub>2</sub>S sensing techniques is imperative for hazard mitigation. While H<sub>2</sub>S sensors have been extensively used in biological systems [13-15], their application in non-biological settings, where the gas is prevalent in high-temperature environments or from industrial processes, remains relatively overlooked [16-18]. Therefore, there is a pressing need for improved H<sub>2</sub>S sensors to detect this gas effectively in diverse non-biological systems.

Nanocrystalline ferrimagnetic materials, a significant category of magnetic materials, find wide-ranging technical applications, including magnetic storage and microwave devices. Their unique structure, comprising two magnetic sub-lattices - tetrahedral and octahedral - and cation distribution, governs their electrical and magnetic properties, making them ideal for gas detection. With both normal and inverse spinel structures, these materials exhibit low dielectric loss, high electrical resistivity, and minimal eddy current losses, making them promising candidates for various applications, including gas sensors [19-29]. Their recent surge in importance is attributed to their distinctive characteristics compared to bulk materials and their versatility across biomedical, hyperthermia, magnetic fluid, catalyst, and gas sensor applications.

Various physical deposition techniques, such as magnetron sputtering, RF sputtering, and pulsed laser deposition, are employed to fabricate thin films [30-

35]. However, these methods come with limitations, such as restricted deposition area, complexity of instruments, material wastage, high operating costs, and the need for cleaning after each deposition. Consequently, the significance of wet chemical processes like chemical vapor deposition, sol-gel, dip coating, electrochemical deposition, and spray pyrolysis has risen, particularly for depositing advanced organic materials like ferrimagnetic materials, addressing the drawbacks of physical methods [36-41].

Wet chemical techniques offer a convenient, affordable means to deposit thin films made of metal oxide materials [42-46]. They ensure uniform deposition without pinholes, which is crucial for nanostructure creation. Controlling preparative parameters, such as complexing agent type, concentration, pH, and deposition temperature, is straightforward. Among these techniques, spray pyrolysis stands out as it is simple, cost-effective, and allows for controlled size, homogeneous grain dispersion, and rapid deposition. Its capability to deposit blended thin films and achieve precise stoichiometry, as well as create porous structures, makes it advantageous for catalytic, gas sensing, and thermal insulation applications.

Ferrimagnetic gas sensors exhibit reversibility, with resistance increasing upon interaction with oxidizing gases and decreasing with reducing gases for n-type materials. Conversely, resistance increases with reducing gases and decreases with oxidizing gases for p-type materials. Key parameters for evaluating gas sensor performance include sensitivity (S), selectivity (K), response time, recovery time, and stability. Sensitivity is calculated as  $(S) = (R_g - R_a) / R_g$ , where  $R_a$  and  $R_g$  are the resistance values before and after gas exposure, respectively. Selectivity is expressed as  $K = S_A / S_B$ , representing the sensor's responses to target gas  $S_A$  and interference gas  $S_B$ . Response time signifies the duration for the sensor to achieve 90% of its maximum resistance change post-exposure to the target gas, while recovery time denotes the time taken for the sensor to regain 90% of its initial resistance. Stability reflects the sensor's ability to maintain consistent performance over a specific duration.

Shimada *et al.* used the spin spray technique to deposit Ni-Zn ferrimagnetic thin films, demonstrating that the spin rotation, which is dominated by crystallographic anisotropy, is responsible for the permeability [47]. Sun *et al.* reported on Sn-substituted Ni-Zn ferrimagnetic thin films made using the sol-gel process. The saturation magnetization was shown to decrease with an increase in Sn concentration [48]. Moreover, Zhenfa *et al.* investigated the impact of annealing temperature on the morphological and magnetic properties of Ni-Zn ferrimagnetic thin films, utilising a straightforward chemical approach [49]. There are few

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