

# Synthesis and Advanced Applications of Transition Metal Oxides

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## Abstract

Transition metal oxide semiconductor materials have gained significant attention in recent years because of their unique electrical, magnetic, and optical features. Transition metal oxides (TMOs) with partially filled d-shells have a wide range of characteristics and play important roles in a variety of technological applications. They are influenced by stoichiometry changes, which alter their physical, chemical, and electrical properties. Controlled synthesis, doping, and post-synthesis treatments can all have an impact on the size, shape, crystal structure, and overall attributes of TMOs. These changes affect applications such as magnetic materials and smart windows. Effective techniques include valence state control, dopants, alloying, structure engineering, nano and defect structuring, and surface modification. This article discusses the synthesis methods, crystal structures, electrical properties, and applications of transition metal oxides, as well as their involvement in cutting-edge technologies such as electronics, optoelectronics, energy storage, and catalysis.

**Keywords:** Transition metal oxides; Synthesis; crystal structure; advanced application; energy storage.

## 1. Introduction

Transition metal oxide (TMO) semiconductor materials are an intriguing group of compounds with distinct electrical, magnetic, and optical properties. These materials are composed of transition metal elements coupled with oxygen, resulting in a variety of chemical compositions and crystal forms. Their importance has expanded dramatically due to advances in materials science and novel applications. TMO, with their distinctive magnetic properties, can be used in spintronics, a science that processes and stores information using electron spin, with the goal of creating faster, more efficient, and higher-density data storage systems. TMO materials are being investigated for usage in next-generation energy storage technologies such as lithium-ion batteries and supercapacitors. TMO are compounds generated when metals combine with oxygen and have a customizable size-shape-crystal structure and characteristics, making them useful in a variety of applications. These TMOs can be modified by optimizing the elemental composition of cationic compounds in the system, which may include changing the metal cation, regulating the metal's oxidation state, doping with various elements, or manipulating the oxygen stoichiometry. Controlling stoichiometry in TMOs is critical for adapting these materials to specific applications in domains such as electronics, energy, and catalysis. For example, iron oxide exists in two distinct phases: ferric oxide ( $\text{Fe}_2\text{O}_3$ ) and ferrous-ferric mixed oxide ( $\text{Fe}_3\text{O}_4$ ), with stoichiometry differences affecting electrical conductivity, magnetic behaviour, and value in magnetic storage and biomedical applications. Copper oxides, such as Cupric Oxide ( $\text{CuO}$ ) and Cuprous Oxide ( $\text{Cu}_2\text{O}$ ), have different properties and applications.  $\text{V}_2\text{O}_5$  is used in batteries, catalysis, and optical coatings, whereas  $\text{VO}_2$  undergoes a unique metal-insulator

transition near room temperature, making it suitable for applications in smart windows, sensors, and thermal regulation systems. TMOs like  $\text{SnO}_2$ ,  $\text{Ag}_2\text{O}$ ,  $\text{TiO}_2$ ,  $\text{ZnO}$ , and  $\text{MnO}_2$  have unique electrical conductivity, transparency, gas-sensing properties, piezoelectric properties, stability, and reactivity. Deviations from ideal stoichiometry can generate defects, change the electronic structure, modify optical characteristics, change conductivity, affect stability, and limit the use of certain metal oxides for specific applications. Controlling the stoichiometry of these oxides is critical for modifying their properties for a variety of technological applications. This article discusses the TMO's synthesis method and its application.

## 2. Synthesis Method of TMO

TMOs offer versatility in synthesis routes, allowing researchers to prepare materials with varying physicochemical attributes such as sol-gel, precipitation, hydrothermal, solvothermal, co-precipitation, Chemical vapor deposition and the Solid States Method.

### 2.1 Co-Precipitation Method

A simple and affordable technique for creating nanomaterials is the precipitation method. An anionic solution containing reducing agents and a cationic solution containing metal salts are involved. When the other solution reduces the hydroxides in the cation solution, precipitates are created. A number of variables, including pH, reaction temperature, stirring speed, salt concentration, and reducing agents, affect these nanoparticles' characteristics.

### 2.2 Sol-Gel Method

A sol-gel synthesis is used to create a variety of materials that are exceedingly pure and homogenous, including ceramics, glasses, thin films, and nanoparticles. Through carefully regulated chemical processes, a precursor "sol" is transformed into a solid substance. Sol-gel synthesis is a potent approach because it allows for precise control over the final material's composition, particle size, porosity, and shape. In a suitable solvent, the compounds comprising the constituents of the desired substance or precursors dissolve to produce a solution, or "sol," which is a colloidal suspension. For consistent precursor dispersion, it is thoroughly agitated. Gelation can be brought about by chemical reactions, temperature changes, or solvent evaporation. The sol changes from a condition resembling liquid to a semi-solid state. To create a porous solid substance known as a "xerogel," the solvent must be removed from the gel using air drying, freeze drying, or supercritical fluid extraction. Remove any leftover organic components from the xerogel by heating it to a high temperature in a controlled environment (such as air, oxygen, or nitrogen) and then start the chemical processes that will result in the production of the final solid substance. Nanoparticles and nanocomposites, ceramic materials like alumina, silica, and titania, thin films and coatings for use in optics, electronics, and catalysis, optical materials like glasses and photonic devices, and biomaterials like bioactive glasses and bio-ceramic are all produced using it. Hence the Sol-Gel process is a chemical procedure that converts a chemical precursor solution into a gel, resulting in solid material. It entails dissolving metal salts or alkoxides in a solvent and generating a homogenous solution via hydrolysis and condensation processes. The solvent is eliminated, resulting in a porous substance that undergoes structural evolution with age. This approach produces uniform, high-purity materials while also providing control over thin films and coatings.

### 2.3 Solvothermal Method

Using solvothermal methods, a range of transition metal oxide semiconductors for photocatalysis, photovoltaic, and electrical devices may be produced. Solvothermal synthesis organic solvents are used,

a reaction media is created by maintaining the precursors and water (aqueous solution) under high pressure. Reactors made of materials that can withstand high pressures and temperatures are frequently made of stainless steel or Teflon-coated autoclaves, allow the reaction to continue for a certain amount of time. The transition metal oxide semiconductor material is created during this period as a result of chemical interactions between the precursors and the water. Cool the reaction vessel gradually until it reaches room temperature. The substance might settle and crystallize inside the solution during this cooling period. Remove the solid result from the solution once it has cooled. Centrifugation or filtration can be used to accomplish this. Using a suitable solvent to rinse the collected solid product to get rid of any impurities or remaining reactants. To get the desired transition metal oxide semiconductor material, dry the product last. Thus, the solvothermal method is a closed-vessel procedure that uses a solvent at high temperatures and pressures to precisely control crystal formation, shape, and size. It is useful for producing high-quality, well-defined crystalline materials, especially for catalysis, nanomaterials, and specific crystalline structures such as layered, spinel, and perovskites. However, it necessitates specialized equipment to manage high temperatures and pressures, potentially making it more complicated and expensive than other approaches.

#### **2.4 Hydrothermal Method**

Hydrothermal synthesis is similar to solvothermal synthesis, but uses water as the major solvent. It is less sophisticated and runs at lower temperatures. Templates and surfactants have an important role in controlling the morphology, structure, and characteristics of synthetic materials. Templates direct crystal and nanoparticle formation, whereas surfactants stabilize colloids, control particle size, and change surface properties. These additives influence morphology, stabilize particles, and modify material characteristics. To maximize their influence on the synthesis process, these additives must be carefully selected and controlled in terms of concentration, temperature, and reaction time. One-dimensional tin oxide nanoparticles can be modified using template-free hydrothermal synthesis by altering reaction time, temperature, solvent volume ratios, and other parameters, which have a substantial impact on SnO<sub>2</sub> nanoparticle morphology.

#### **2.5 Solid State Method**

Many different types of materials, including ceramics, semiconductors, and complicated chemicals, are produced using the solid-state synthesis technique. This process includes the high-temperature reaction of powdered solid-state precursors. The requisite elements and compounds required to create the target material in the form of powders or crystals are found in high-purity solid-state precursors. By using a mortar and pestle, thoroughly combine the powders to ensure that the components are distributed evenly. A crucible made of alumina (Al<sub>2</sub>O<sub>3</sub>) or another material that can resist high temperatures is used to hold the combined particles. After that, the crucible is heated in a furnace or oven. It is used to gradually eliminate any moisture or volatile contaminants from the mixture by heating it to a low temperature (below 200 °C). In order to start chemical reactions, the temperature is raised to a reasonable level (usually between 400°C and 900°C). At this point, intermediate compounds may occur. Crystal development will be encouraged and the creation of the desired product will be made easier with further heating to higher temperatures (typically exceeding 1000°C). Various controlled atmospheres, including air, oxygen, nitrogen, argon, or a reducing atmosphere, can be used to conduct the synthesis. This approach entails a direct interaction between solid reactants at high temperatures, usually without a solvent. The combination is blended in the appropriate amounts, then grinding is utilized to increase reactivity. The mixture is then heated to a specified temperature, generating chemical interactions between the components. Post-reaction

annealing or sintering at specified temperatures may be used to improve crystallinity and characteristics. This approach enables the targeted manufacture of transition metal oxides for a variety of applications in electronics, catalysis, and material science.

### **2.6 Chemical vapor deposition (CVD)**

Chemical vapor deposition (CVD) technique is used to produce thin films and coatings of a variety of materials, including semiconductor transition metal oxide compounds. For the manufacture of semiconductor devices and other applications, CVD is a good choice since it allows for fine control over layer thickness, composition, and crystalline structure.

Select appropriate precursors for the deposition of the transition metal oxide. These precursors are often volatile substances that may vaporize and break down at high temperatures, frequently metalorganic substances or metal halides. To move the vaporized precursors to the substrate in the CVD reactor, choose a carrier gas (such as nitrogen or hydrogen). Heat the chosen precursors in a vaporization chamber to cause them to vaporize. In order to achieve appropriate vaporization, the temperature and pressure parameters must be managed. To get the right film composition, carefully combine the vaporized precursors with the carrier gas. Bring the carrier gas that is loaded with precursors to the deposition chamber so that it may interact with the heated substrate there. The precursor gases interact chemically on the surface of the substrate, which causes transition metal oxide material to deposit there. The selected precursors and the reaction circumstances will determine the precise reactions. Heat the substrate to a level that will allow the required crystal structure to develop. To achieve the necessary film characteristics, temperature management is essential. A thin coating of the transition metal oxide semiconductor material forms on the substrate. Precursor flow rates, temperature, and reaction times are a few examples of the variables that affect the deposition rate and film thickness. For the fabrication of semiconductor devices (such as thin-film transistors, capacitors, and resistors), photovoltaic (such as thin-film solar cells), gas sensors, transparent conductive oxide coatings, and catalytic materials, CVD is used to deposit transition metal oxide semiconductor materials.

### **3. Inter-relation between Structure-Composition and Morphology**

TMOs are flexible materials with a variety of crystal forms, which determine their conductive and catalytic capabilities. Imperfections such as vacancies and dopants can change electronic properties, with oxygen vacancies regulating conductivity and influencing applications such as Li-battery technology. TMOs can have different shapes, including nanowires, nanosheets, and nanoparticles, which are important in catalysis and energy storage. Surface characteristics such as energy distribution and active sites influence interactions in catalysis and sensing. The electrical and magnetic properties of TMOs vary depending on their composition, with rare earth elements or additions changing electronic configurations and magnetic behaviours. Electron configurations and charge mobility are critical factors in semiconductor and electrical device applications. TMOs' various magnetic behaviours are intertwined with structural nuances and elemental constituents, which are critical in spintronic applications. Iron oxide exhibits different magnetic properties, which are critical in magnetic data storage applications. TMOs catalytic efficiency is determined by surface characteristics and redox reactions, which shape chemical processes. Band structures and energy gaps dictate optical characteristics, which are critical in optoelectronic devices and sensors. Understanding these linkages enables personalized designs for specific uses.

#### 4. Different Methods for Analysis of TMOs

Understanding and harnessing these multifaceted properties of TMOs are crucial for designing and developing advanced materials for applications spanning electronics, energy storage, catalysis, sensing, and beyond. Their broad electronic and magnetic behaviors, ranging from insulating to conducting and varied magnetic properties, are pivotal in applications like storage and spintronics. Some TMOs display high conductivity and semiconducting traits, vital in sensors and optoelectronics. Remarkable catalytic properties and redox activity make them key in industrial processes and energy conversion. Optical versatility aids in photovoltaics and sensors. Morphological features influence reactivity, while tunable properties allow tailored applications in research and industry across electronics, energy, catalysis, and sensing.

The different methods of analysis for TMOs for studying various properties like structural, optical, chemical composition and bonding, morphology, electrochemical, electrical, and magnetic. Structural properties measured by XRD, neutron scattering, atom probe tomography, optical properties by UV-visible-near infra-red spectroscopy, photoluminescence, chemical composition and bonding by FTIR, NMR, Raman spectroscopy, Mossbauer spectroscopy, EDX analysis, XPS analysis, morphology by SEM/TEM/AFM, electrochemical properties by impedance spectroscopy, surface plasmon resonance, electrical and magnetic properties analysis by MRI, quantum sensing etc.

#### 5. TMO's Applications

To address energy and environmental sustainability issues caused by fossil fuel depletion and greenhouse gas emissions, eco-friendly and efficient materials such as 2D transition metal oxide (TMO) are essential. However, their low electronic conductivity limits its usage in energy-related applications. TMOs are high-performance materials used in energy storage, sensors, water purification, CO<sub>2</sub> reduction, electronics, and other applications. Transition metal oxides are valued for their unique surface and physicochemical features in multiphase catalysis. Transition metals conduct pseudocapacitive reactions, which cause electrochemical phenomena throughout the electrode, increasing capacitance and boosting energy density. These materials' dispersed reactivity illustrates their broad uses and complicated features in catalytic processes. TMOs are critical electrodes in lithium-ion batteries, allowing charge storage via ion insertion / de-insertion and ion diffusion mechanisms. Efficient ion diffusion is critical for optimal battery performance, with composite electrodes containing multiple metals surpassing single metal electrodes in capacity, stability, and charging/discharge rates. Various Applications of TMO Materials depicted in figure 1.



**Figure 1. Various Applications of TMO Materials**



The lithium-ion battery, a high-performance rechargeable secondary battery, remains a focus in modern battery technology. Researchers explore various materials, including nickel oxides, carbon, and graphene materials, for improved lithium battery efficiency. In the realm of water treatment, the escalating issues of population growth, industrial expansion, and water pollution demand effective solutions. Transition metal oxides (TMOs) have gained attention for their versatile composition and catalytic properties. Researchers explore binary, ternary, quaternary, and heterostructure TMOs for various pollutants, utilizing photocatalysis, adsorption, antimicrobial action, persulfate/peroxy-mono-sulfate activation, and Fenton-like processes. Transition metal oxides also play a vital role in healthcare applications due to their electrocatalytic activity and cost-effectiveness. Metal oxide nanocomposites are widely employed for detecting emerging biomarkers. developed a biosensor platform for cholesterol detection using thionine and  $\text{Cu}_2\text{O}$  nanomaterials, demonstrating high sensitivity and a broad sensing range. In conclusion, the integration of transition metal oxides in energy, water treatment, and healthcare applications offers promising avenues for addressing contemporary challenges. Research efforts focus on enhancing the properties of these materials, leading to more efficient and sustainable solutions.

### 5.1 Applications in Electronics

Transition metal oxides (TMOs) are versatile semiconductor materials with unique features that make them suitable for a wide range of applications, including field-effect transistors (FETs), flexible and transparent devices, sensors, memory devices, and high-speed electronics. Transparent touch screens, flexible displays, and wearable electronics all make use of flexible FETs like indium tin oxide (ITO). TMOs are also utilized in resistive switching memory, which are formed by depositing zinc oxide (ZnO) or indium zinc oxide (IZO) on flexible and transparent substrates. They can function as electrodes for electrical measurements in biomedical equipment, reduce heat input and glare in building windows, regulate heat and light in electrochromic devices and smart windows, and improve solar energy conversion efficiency by collecting generated electrical current while allowing sunlight to pass through. TMOs are also employed in solar cells and photovoltaic systems because they provide electrical conductivity for touch sensing, facilitate light transmission, and enable touch sensitivity. TMOs have a diverse variety of applications due to their unique combination of optical transparency and electrical conductivity.

### 5.2 Applications in Sensors

TMOs are commonly employed in gas sensors to detect gases such as CO, NO<sub>2</sub>, and CH<sub>4</sub>. They are extremely sensitive, selective, and stable, making them ideal for environmental monitoring, industrial safety, and automotive applications. ZnO and TiO<sub>2</sub> are examples of TMOs that can be utilized as humidity sensors in climate control, agriculture, and weather monitoring. They also exhibit consistent resistance variations in reaction to temperature changes, making them ideal for temperature sensors in electrical devices, industrial processes, and automotive applications. TMOs can be programmed to detect specific substances or ions in solution, making them helpful for water quality monitoring, chemical analysis, and medical diagnosis. They are also employed in biosensors that detect infections, biomarkers, and other biological substances, as well as pressure sensors for medical equipment, industrial automation, and automobiles. TMOs having photodetector properties, such as ZnO, are employed in light sensors, photodiodes, and photovoltaic systems, expanding their applications in photovoltaic systems, optical communication, and image sensing.

### 5.3 Applications in Optoelectronics

Transition metal oxide (TMO) semiconductor materials are widely employed in photodetectors and photovoltaics due to their distinct electrical and optical properties. Zinc oxide, titanium dioxide, and

tungsten oxide are capable of sensing visible and ultraviolet light, making them ideal for applications such as flame detection, UV imaging, and radiation monitoring. Vanadium dioxide and molybdenum disulfide are good for infrared photodetectors, which are utilized in thermal imaging systems, remote sensing, and night vision devices. Indium tin oxide (ITO) serves as transparent conducting electrodes in LEDs, allowing light to pass through the structure.

#### **5.4 Applications Water splitting**

Transition metal oxide semiconductor materials (TMOs) play an important role in photo-electrochemical (PEC) cells, notably water splitting, which is required for the production of renewable hydrogen and solar fuel. When exposed to sunlight, TMOs such as bismuth vanadate, hematite, and titanium dioxide absorb photons and generate electron-hole pairs, so commencing the water splitting mechanism. TMOs absorb visible light, allowing for more efficient solar energy usage and the development of PEC systems that do not rely entirely on UV radiation. The reduction of protons at the photo-anode in PEC cells employing TMOs generates hydrogen gas ( $H_2$ ), a clean and renewable energy source. In addition to hydrogen, PEC cells may produce oxygen gas ( $O_2$ ), a beneficial byproduct with numerous applications in industrial and medical contexts.

#### **5.5 Application in Dye-Sensitized Photovoltaic Cells (DSSC)**

Dye-sensitized organic photovoltaic cells are a hybrid structure that conducts electrons via an electrolyte solution with organic dyes and a titanium dioxide nanocrystalline layer. These cells are commercializing and have a power conversion efficiency of approximately 11%, allowing for efficient ion movement in the electrolyte.

#### **5.6 Applications Energy Storage**

##### **5.6.1 Lithium-ion batteries**

Transition metal oxide (TMO) semiconductor materials are widely employed in energy storage, particularly lithium-ion batteries. These materials have a significant impact on the performance and capacity of Li-ion batteries, which are increasingly employed for a variety of applications such as portable electronics, electric cars, and grid-scale energy storage. Lithium cobalt oxide ( $LiCoO_2$ ), lithium manganese oxide ( $LiMn_2O_4$ ), and lithium iron phosphate ( $LiFePO_4$ ) are examples of TMO cathodes used in Li-ion batteries. High-energy-density Li-ion batteries used in electric vehicles (EVs) and massive energy storage systems (MESS) use TMO cathode materials such as lithium nickel cobalt manganese oxide (NCM) or lithium nickel cobalt aluminium oxide (NCA).  $LiFePO_4$  is noted for its high power and rapid charge/discharge rates, making it ideal for usage in power tools, portable gadgets, and electric vehicles. Molybdenum disulfide ( $MoS_2$ ) and titanium dioxide ( $TiO_2$ ) are also employed as anode materials, which offer more stability and safety than traditional carbon-based anodes.

##### **5.6.2 Supercapacitors**

Transition metal oxide (TMO) semiconductor materials, especially in supercapacitors, provide high power density, rapid charge/discharge rates, and long cycle life in energy storage. These materials, including ruthenium oxide ( $RuO_2$ ), manganese oxide ( $MnO_2$ ), and cobalt oxide ( $Co_3O_4$ ), can be used in regenerative braking systems in hybrid and electric vehicles, energy harvesting devices, wireless sensors, remote monitoring systems, and low-power electronics. To address peak power demands, electrical systems use supercapacitors with TMO electrodes, which swiftly release stored energy during moments of high demand. TMO-based supercapacitors can also be used to provide backup power in electronic devices such as real-time clocks, embedded systems, and solid-state drives. TMO-based batteries and

supercapacitors can be used to generate hybrid energy storage systems (HESS). Combining these two features allows for long-term energy storage and high-power bursts, making them excellent for uninterruptible power supply (UPS) and electric grid stabilization. Overall, TMO semiconductor materials present potential possibilities for energy storage in a variety of applications.

### 5.7 Applications Catalysts

Transition metal oxides (TMOs) are important catalysts in a variety of chemical processes due to their distinct electrical and structural properties. They provide an alternative reaction channel with lower activation energy, allowing reactions to proceed more quickly and easily. TMOs play an important role in a variety of activities, including energy generation, environmental protection, and the synthesis of valuable chemicals and minerals. Their surfaces have the ability to adsorb and activate reactant molecules, weakening internal linkages during the adsorption process. They also give places for reactants to cluster and interact, with metal cations in various oxidation states being found at these spots. These cations can modify their electron configurations and form transient connections with reactants, allowing them to shift the reaction's direction with less activation energy.

TMO catalysts accelerate reactions at a given temperature by lowering activation energy and increasing rate of reaction, which is useful in industrial activities that require high efficiency and throughput. TMOs are used in fuel cells to perform the oxygen reduction process (ORR) at the cathode, cutting the overall cost of fuel cell systems while increasing the catalytic activity of platinum and other precious metal catalysts. They also act as catalysts in supercapacitors, rechargeable batteries, and other energy storage devices, enhancing performance and charge/discharge dynamics.

### 6. Conclusion

Transition metal oxides (TMOs) have numerous uses in electronics, energy storage, catalysis, and sensing. They are optimized by controlled synthesis, temperature modulation, and crystal structure engineering. TMO characteristics are being refined by continuing research, which will result in significant advancements in materials science and technology. Future research will focus on novel synthesis processes and advanced temperature control, with future technologies such as 2D and 3D TMOs having the potential to revolutionize energy storage and demonstrate advances in materials science and technology. Transition metal oxide semiconductor materials are important for their distinct features and applications in electronics, optoelectronics, energy conversion, and environmental remediation. They are positioned to have a big impact on memory technology and associated applications in the future. TMOs used as essential building blocks in the creation of cutting-edge electronic gadgets, renewable energy sources, and environmental technologies because of their distinctive electronic structure and customizable characteristics. Their continued research and development are intended to accelerate technical developments and contribute to a more sustainable future.

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