

Investigation of Mixed Metal Oxide Bilayers in Photoelectrochemical Water Splitting: Synthesis and Functional Properties

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ABSTRACT

The global annual energy consumption indicates a pressing necessity to improve efficient renewable energy technologies. PEC water splitting represents a highly promising method for generating renewable energy through solar power. Recently, mixed metal oxide bilayered thin films have emerged as promising materials for PEC water splitting, due to their tunable optoelectronic behavior, enhanced charge separation, and improved stability of the films. This document offers an in-depth analysis of synthesis techniques, structural and functional properties, and the photocatalytic energy conversion efficiency of double-layer metal oxide materials, such as BaZrO₃/Cu₂O, SrTiO₃/Cu₂O, and SrTiO₃/CeO₂. The discussion subsequently centers on the latest advancements in material science, including selection processes, fabrication techniques, and improvements in PEC devices. The paper's final section outlines a series of challenges that could impact the effectiveness of these systems, along with potential solutions. Additionally, it discusses future inquiries that might enhance the applicability of these systems in large-scale hydrogen production.

Keywords- Photoelectrochemical (PEC), Metal Oxide, Water-splitting, Physical Vapor Deposition (PVD), Sol-gel.

I. INTRODUCTION

1.1. Renewable Energy and PEC Water Splitting

The increasing reliance on fossil fuels in the global market has sparked significant concern regarding the need for environmentally friendly and sustainable renewable energy sources. Hydrogen is increasingly promoted as a secondary energy carrier derived from renewable energy sources due to its clean combustion with water and its high energy density. This positions hydrogen as a promising option for achieving sustainable energy solutions. PEC water splitting represents a highly promising method for generating hydrogen through the electrolysis of water, utilizing sunlight as the energy source (Samuel et al., 2020). The PEC water splitting approach presents a large-scale solution, and with its straightforward setup, the effective use of abundant sunlight renders it a highly attractive option for hydrogen generation. The PEC water-splitting systems consist of

semiconductor-based photoelectrodes that harness light to facilitate the water-splitting process by delivering the necessary electrochemical potential. Nonetheless, various obstacles limit their implementation, such as the need for highly efficient and stable photoelectrodes under operational and cost-effective conditions. This necessity has driven extensive investigation into semiconductor-based systems, particularly those involving mixed metal oxides. Due to their adaptable electronic, optical, and chemical properties, mixed metal oxides are considered promising materials for photoelectrodes. The introduction of these materials can be strategically designed to achieve the desired bandgap, enhance the conductivity of selected charges, and boost the catalytic potential. Moreover, due to the structural durability of mixed metal oxides in harsh PEC environments, this compound is well-suited for extended applications. To enhance the performance of structures, experts have explored the concept of utilizing two materials with distinct characteristics simultaneously,

known as bilayer structures, which comprise two different metal oxide layers. The efficiency of water splitting through photocatalytic methods has seen significant enhancement across various mixed metal oxide systems, including bilayer architectures such as BaZrO₃/Cu₂O, SrTiO₃/Cu₂O, BaTiO₃/Cu₂O, and SrTiO₃/CeO₂ (Yang et al., 2017). These systems utilize the unique properties of each layer to improve light absorption, charge separation, and surface catalytic activity. For instance, integrating a wide-bandgap oxide like SrTiO₃ with a narrow-bandgap material like Cu₂O allows efficient solar spectrum utilization while maintaining stability. Similarly, BaTiO₃ and BaZrO₃ contribute ferroelectric and dielectric properties, facilitating charge separation and reducing recombination losses.

The creation of these bilayered systems as outlined necessitates the application of advanced technological techniques such as pulsed laser deposition, chemical vapor deposition, and sol-gel processes. These techniques allow the filmmaker to attain precise regulation of the film thickness, material components, and interface quality, all of which are essential for enhancing PEC performance. Furthermore, the application of doping and surface modification techniques enhances the electronic properties of the bilayers, leading to greater efficiency (Gao et al., 2022). Nonetheless, various obstacles remain apparent in the creation of mixed metal oxide bilayers for effective PEC water splitting. Concerns include the durability of substances under prolonged light exposure, the movement of charges at boundaries, and the expansion of manufacturing processes.

1.2. Role of Mixed Metal Oxide Bilayers

Metal oxides have been recognized as a crucial basis for creating materials essential in photoelectrochemical PEC, owing to their chemical properties, accessibility, and suitability for photoelectrocatalytic energy processes that utilize solar light. All these characteristics come together to render them ideal for generating hydrogen via water splitting, promoting sustainable utilization. While extensive studies have focused on individual metal oxide substances, the difficulties related to light absorption, charge carrier recombination, and catalytic conversion necessitate improved approaches (Ikram et al., 2021).

To tackle these challenges, systems composed of two distinct metal oxides layered together seem to offer the most effective solution. Indeed, the compatibility of the materials utilized in the preparation of the mixed metal oxide bilayers enhances their light absorption properties and boosts charge separation performance. The blend of materials offers a chance to adjust the electronic band structure, enhancing charge transfer at the interface while reducing recombination losses. This collaboration not only improves the effectiveness of PEC but also expands the spectrum of light absorption, thereby boosting the overall quantum

conversion efficiency. Recent advancements in thin films showcase the stacking of mixed metal oxide bilayers such as BaZrO₃/Cu₂O, SrTiO₃/Cu₂O, and SrTiO₃/CeO₂. BaZrO₃, which exhibits superior stability and a suitable bandgap in comparison to Cu₂O, improves the visible light absorption efficiency of the developed bilayer for hydrogen production (Antončík et al., 2020). Similarly, employing SrTiO₃ alongside Cu₂O or CeO₂ enhances their electronic and optical properties, leading to significant advancements in PEC performance metrics. These bilayer systems demonstrate enhanced durability, addressing one of the primary challenges associated with single-layer materials in PEC applications. Research in PEC could lead to future breakthroughs in sustainable hydrogen production, and exploring mixed metal oxide bilayers may pave the way for advancements in renewable energy sources.

II. SYNTHESIS OF MIXED METAL OXIDE BILAYERS

2.1. Thin Film Deposition Techniques

The synthesis of bilayered thin films requires precise control over material composition, thickness, and interface quality. These parameters are critical in ensuring the desired electronic, optical, and catalytic properties essential for efficient photoelectrochemical water splitting. Techniques such as physical vapour deposition (PVD), chemical vapour deposition (CVD), atomic layer deposition (ALD), and pulsed laser deposition (PLD) enable the fabrication of high-quality bilayers with tunable properties. Advanced control over deposition conditions ensures uniformity, minimizes defects, and optimizes the interaction between the mixed metal oxide layers, enhancing their performance in PEC applications (Jang & Lee, 2019).

2.1.1. Physical Vapor Deposition (PVD)

Physical Vapor Deposition (PVD) encompasses advanced techniques like sputtering and electron beam evaporation, which are pivotal in fabricating high-quality bilayered thin films for photoelectrochemical (PEC) water splitting applications. These methods are renowned for their ability to produce uniform and adherent films with precise control over composition and thickness, essential for optimizing the functional properties of mixed metal oxide bilayers (Pedro & feil, 2016). Sputtering involves ejecting material from a target source onto a substrate using energetic ions, facilitating the deposition of dense and smooth films.

This technique is highly versatile, enabling the deposition of a wide range of metal oxides and their mixtures with excellent stoichiometric control. Electron beam evaporation, on the other hand, employs a focused electron beam to vaporize a target material, which then condenses onto the substrate to form a film. This method is particularly advantageous for achieving high-purity and defect-free layers. The precise control over deposition parameters in both techniques allows

researchers to tailor the interfacial properties of bilayers, such as band alignment and charge transport characteristics, thereby enhancing their PEC performance. PVD processes also enable scalability and integration with various substrates, making them indispensable in developing advanced bilayer systems for sustainable hydrogen production through PEC water splitting.

2.1.2. Chemical Vapor Deposition (CVD)

Chemical Vapor Deposition (CVD) is a highly versatile and widely adopted technique for fabricating thin films and complex bilayer structures. The process involves the chemical reaction of vapor-phase precursors on a heated substrate, leading to the deposition of high-purity materials. CVD is particularly advantageous for creating uniform films with excellent conformality, even on substrates with intricate geometries. This characteristic makes it ideal for synthesizing mixed metal oxide bilayers, which often require precise control over thickness and composition to optimize their photoelectrochemical (PEC) properties. By fine-tuning parameters such as temperature, pressure, and precursor flow rates, CVD facilitates the deposition of dense, defect-free layers with tailored functional properties (Wang & Yushin, 2015). Furthermore, its compatibility with various metal oxide systems, including BaZrO₃/Cu₂O and SrTiO₃/CeO₂, underscores its utility in advancing PEC water-splitting applications. This method ensures enhanced interfacial quality, crucial for efficient charge separation and transport in bilayered semiconductors.

2.1.3. Sol-Gel Processing

Sol-Gel Processing is an effective and cost-efficient technique for synthesizing mixed metal oxide bilayers, crucial for enhancing photoelectrochemical water splitting efficiency. This method involves the preparation of precursor solutions that contain metal salts, which are converted into metal oxide films through a chemical reaction. In the first stage, a sol is formed by dissolving metal salts in a solvent, creating a colloidal suspension. The sol is then applied to a substrate using techniques such as spin-coating or dip-coating. These methods ensure uniform film deposition. Following the coating, the film is subjected to an annealing process at high temperatures, which promotes the crystallization of the metal oxides, improving their structural and functional properties. Annealing also helps remove organic residues and improves the overall film adhesion to the substrate (Pattnaik et al., 2023). The sol-gel process offers precise control over film thickness, composition, and morphology, making it an ideal choice for fabricating mixed metal oxide bilayers with enhanced performance in photoelectrochemical applications.

2.1.4. Pulsed Laser Deposition (PLD)

Pulsed Laser Deposition (PLD) is a widely utilized thin-film deposition technique that plays a crucial role in the synthesis of high-quality films for various applications, including photoelectrochemical

water splitting. PLD is particularly advantageous for fabricating complex metal oxide structures because it provides precise stoichiometric control over the deposited material. In this process, a high-energy laser pulse is directed onto a target material, causing it to vaporize and form a plasma plume (Wang et al., 2020). This plume then deposits onto a substrate, where it condenses to form a thin film. The process allows for the deposition of a variety of materials, including mixed metal oxide bilayers, which are often employed in photoelectrochemical devices due to their excellent electronic and optical properties. PLD is favored for its ability to produce highly uniform and reproducible films with controlled thickness, composition, and structure, making it an essential technique for the development of efficient materials for water splitting applications.

2.2. Surface Modification and Interface Engineering

Investigating mixed metal oxide bilayers in photoelectrochemical (PEC) water splitting is a promising area of research aimed at enhancing the efficiency of hydrogen production from water using solar energy. A crucial aspect of this research is the optimization of surface modification and interface engineering, which significantly influences the performance of the photoelectrode materials. The use of surface texturing, interface engineering, and doping as techniques for improving the functionality of mixed metal oxide bilayers plays a vital role in achieving high efficiency in PEC water splitting.

Surface Texturing: Enhancing Light Absorption

Surface texturing is an essential strategy for improving the light absorption capabilities of photoelectrodes used in PEC water splitting. The process involves creating nanoscale or microscale patterns on the material's surface, which reduce light reflection and increase light trapping. This is particularly important in photoelectrochemical systems, where light absorption directly correlates with the efficiency of the water splitting reaction. By minimizing light reflectance, textured surfaces ensure that more photons are absorbed by the photoelectrode, leading to a higher generation of charge carriers (electrons and holes) that are essential for the water splitting reaction (Klotz et al., 2017).

In addition, surface texturing enhances the optical path length of incoming light within the material, promoting light scattering and ensuring better light utilization. This is particularly beneficial for photoelectrodes with limited thickness or low absorption coefficients. Various surface texturing techniques, such as the creation of nanostructured surfaces, mesoporous films, or micro-/nano-patterned layers, have been employed to enhance the performance of photoelectrodes made from mixed metal oxides. These surface modifications contribute significantly to the overall efficiency of the PEC system.

Interface Engineering: Optimizing Charge Transfer

Interface engineering plays a pivotal role in optimizing the charge transfer between the different

components of the mixed metal oxide bilayer. In a bilayer system, one layer typically acts as a light absorber, while the other layer functions as a catalyst for the oxygen or hydrogen evolution reaction. Effective charge transfer at the interface between these layers is critical to minimize energy losses due to charge carrier recombination, which can reduce the overall performance of the photoelectrode.

To reduce recombination losses, the interface must be engineered to ensure efficient electron and hole separation and transport. This can be achieved through various methods, such as controlling the crystal structure, morphology, and alignment of the electronic bands of the two layers (Santbergen et al., 2019). Interface engineering can also involve the introduction of buffer layers or conductive materials that facilitate the smooth flow of charge carriers between the layers. By optimizing the interface, the efficiency of charge transfer is enhanced, which reduces the likelihood of recombination and maximizes the photogenerated current for water splitting.

Doping: Improving Conductivity and Bandgap Alignment

Doping is a technique used to modify the electronic properties of mixed metal oxide layers. By introducing foreign atoms (dopants) into the crystal lattice of the metal oxides, the conductivity and electronic structure of the material can be improved, leading to better performance in PEC water splitting. Dopants can alter the carrier concentration, enhance the electronic conductivity, and modify the material's bandgap, all of which are crucial factors in the performance of the photoelectrode. One of the key advantages of doping is its ability to improve the alignment of the energy bands between the bilayer components. The bandgap of each layer must be carefully tailored to ensure optimal absorption of sunlight and efficient charge transfer. Dopants can help achieve this alignment by modifying the valence and conduction bands of the oxide materials, ensuring that both layers work synergistically for maximum photoelectrochemical efficiency. Furthermore, doping can also help reduce defects in the crystal structure, often sites for electron-hole recombination. By improving the conductivity and bandgap alignment, doping enhances the overall functionality of the mixed metal oxide bilayer for PEC water splitting.

III. STRUCTURAL AND FUNCTIONAL PROPERTIES

3.1. Crystallinity and Morphology

Crystallinity and morphology are crucial factors that determine the photoelectrochemical (PEC) performance of mixed metal oxide bilayers in water splitting applications. The crystal structure of the bilayers influences the electronic properties, charge carrier mobility, and the efficiency of charge separation

and transfer processes, all of which are key to enhancing PEC performance. The crystallinity of the mixed metal oxides can be analyzed using X-ray diffraction (XRD), which provides detailed insights into the phase purity, orientation, and degree of crystallinity of the synthesized bilayers (Pudkon et al., 2020). XRD patterns help to confirm the presence of specific crystal phases and reveal any structural distortions that might affect the overall functionality. Surface morphology is another critical factor, as it influences the interaction between the catalyst and the electrolyte, as well as the availability of active sites for the electrochemical reaction. Scanning electron microscopy (SEM) is often employed to examine the surface morphology, providing high-resolution images of the bilayer surface to evaluate the size, distribution, and shape of particles or nanostructures (Ebnesajjad, 2011).

Atomic force microscopy (AFM) further complements this analysis by offering three-dimensional topographic information, which helps in assessing the surface roughness and the uniformity of the bilayers. A combination of these techniques enables a comprehensive understanding of the structural properties, allowing for optimization of the bilayers for superior PEC performance in water splitting reactions (Wang et al., 2019).

3.2. Bandgap Tuning and Optical Properties

Mixed metal oxide bilayers have garnered significant attention for their tunable bandgap properties, which enable them to absorb light across a broader range of the solar spectrum, a crucial factor for enhancing the efficiency of photoelectrochemical (PEC) water splitting. The ability to adjust the bandgap through material selection and structure makes these bilayer systems ideal candidates for harvesting both visible and ultraviolet (UV) light. For instance, the BaZrO₃/Cu₂O bilayer exhibits a bandgap of approximately 2.3 eV, making it particularly effective for absorbing visible light, which accounts for a substantial portion of the solar spectrum (Choudhary et al., 2012). This characteristic enhances the material's ability to capture sunlight for photoelectrochemical reactions, improving the overall efficiency of water splitting processes. On the other hand, the SrTiO₃/CeO₂ bilayer showcases a bandgap greater than 3 eV, which is ideal for absorbing UV light.

The UV absorption capacity is advantageous for promoting the photocatalytic processes in PEC systems, especially under high-energy photon excitation. Combining materials with distinct bandgap properties, mixed metal oxide bilayers can harness a wide range of light energy, enhancing their photocatalytic efficiency. The tunability of these bandgaps offers a pathway to optimizing the photoelectrochemical performance of mixed metal oxide bilayers, making them promising candidates for sustainable energy conversion technologies such as water splitting (Krishnan et al., 2023).

IV. PHOTOELECTROCHEMICAL PERFORMANCE

4.1. Key Performance Metrics

In the investigation of mixed metal oxide bilayers for photoelectrochemical (PEC) water splitting, understanding key performance metrics is essential for evaluating the efficiency of the system. These metrics not only provide insight into the material's ability to convert solar energy into chemical energy but also help to identify opportunities for enhancing the system's functionality. Photocurrent Density is a crucial parameter that reflects the rate of photo-induced electron generation upon light absorption by the photoelectrode. It is indicative of the efficiency of the material in converting absorbed photons into electrical energy (Gawęda et al., 2009). High photocurrent density is often associated with superior light absorption and charge separation, essential characteristics for an effective PEC water splitting system. Incident Photon-to-Current Efficiency (IPCE) quantifies the efficiency with which incident photons are converted into current. It provides a measure of the material's ability to harness solar energy, which is critical for optimizing the overall performance of photoelectrodes (Krishnan et al., 2023). Higher IPCE values suggest improved efficiency in the light-to-electrical energy conversion process, making it a significant factor in assessing the performance of mixed metal oxide bilayer structures. Hydrogen Evolution Rate (HER) is a vital metric for evaluating the system's capability to produce hydrogen gas, which is the ultimate goal of water splitting. A high HER demonstrates the effectiveness of the material in catalyzing the hydrogen evolution reaction, making it a key indicator for assessing the practical application of mixed metal oxide bilayers in sustainable energy production (Li et al., 2020).

4.2. Mechanisms Enhancing PEC Performance

In the investigation of mixed metal oxide bilayers for photoelectrochemical (PEC) water splitting, several mechanisms contribute to enhancing their performance, driving their efficiency in converting solar energy into hydrogen fuel. One of the primary mechanisms is light absorption, where narrow-bandgap materials play a critical role. These materials, typically in the range of 1.0–2.0 eV, effectively absorb a broader spectrum of sunlight, including the visible and near-infrared regions, leading to an increase in the overall energy conversion efficiency (Pudkon et al., 2020). This expanded absorption range helps optimize the use of solar energy for water splitting. Another vital mechanism is charge separation at the heterojunction interface. Mixed metal oxide bilayers are often engineered with a combination of materials that form a heterojunction, where the alignment of energy levels enhances the separation of photogenerated electron-hole pairs. This efficient charge separation minimizes recombination losses, ensuring that the charges are directed toward the respective cathode and anode, which is crucial for

sustaining the water splitting reaction. Furthermore, oxygen evolution catalysis by wide-bandgap oxides is fundamental to the PEC process. Wide-bandgap oxides, such as titanium dioxide (TiO₂), are employed as catalysts at the anode, facilitating the oxygen evolution reaction (OER). These materials, despite their lower light absorption, are effective in accelerating the OER due to their excellent stability and high catalytic activity under light irradiation, improving the overall PEC performance for sustainable hydrogen production (Li et al., 2023).

V. CHALLENGES AND FUTURE DIRECTIONS

5.1. Challenges

One significant challenge is the presence of interface defects between the layers of the bilayer structure. These defects can severely hinder the efficient transport of charge carriers, essential for PEC performance. The mismatch in lattice structures or poor adhesion between the layers can lead to an increase in recombination of photogenerated charge carriers, thereby reducing the overall efficiency of the system. Such defects undermine the effectiveness of the photoelectrode in water splitting applications (Liu et al., 2021). Scalability is another major challenge, especially when attempting to fabricate large-area mixed metal oxide bilayers with uniform properties. Achieving consistency in the material properties, such as thickness, composition, and crystallinity, becomes increasingly difficult as the size of the film increases. The non-uniformity of the bilayer can result in performance degradation, making it challenging to scale the technology for industrial applications (Baade & Wood, 2021). Finally, the cost associated with deposition techniques, particularly Pulsed Laser Deposition (PLD), is a limiting factor for widespread adoption. PLD, although effective for achieving high-quality thin films, is an expensive technique due to the high equipment cost, the need for precise control over deposition conditions, and the use of high-purity precursors. Reducing costs while maintaining high performance is crucial for the practical application of mixed metal oxide bilayers in PEC water splitting (Wang et al., 2019).

5.2. Future Directions

In the pursuit of enhancing the performance of photoelectrochemical (PEC) water splitting systems, future research on mixed metal oxide bilayers offers promising directions. One critical avenue is the development of advanced deposition techniques. These techniques should aim to be cost-effective and scalable to facilitate large-scale synthesis of mixed metal oxide bilayers, ensuring that the materials can be used in practical applications. Techniques such as atomic layer deposition (ALD) or pulsed laser deposition (PLD) could be optimized for this purpose to achieve uniform, high-quality thin films on a variety of substrates. Another

promising direction is interface engineering, where molecular dynamics simulations can be employed to study and optimize the interfaces between the different layers of the bilayer. Understanding the atomic and electronic structure of these interfaces will be key to improving charge transfer and minimizing energy losses, ultimately enhancing the efficiency of water splitting reactions. The integration of co-catalysts, such as platinum (Pt) or ruthenium oxide (RuO₂), is another future focus. These catalysts can play a crucial role in improving the reaction kinetics at the electrode surface, promoting faster hydrogen and oxygen evolution reactions. Additionally, exploring novel combinations of mixed oxide systems like ZnO/TiO₂ or BiVO₄/WO₃ could lead to new materials with enhanced photocatalytic properties, potentially providing better stability, light absorption, and charge separation for more efficient PEC water splitting.

VI. CONCLUSION

In conclusion, mixed metal oxide bilayer systems have emerged as a significant advancement in the field of photoelectrochemical (PEC) water splitting, providing a promising pathway to efficient and sustainable hydrogen production. These bilayer systems capitalize on the synergistic effects of combining different metal oxides, each contributing unique properties such as enhanced light absorption, improved charge separation, and increased stability. As a result, they outperform traditional single-material systems in terms of overall efficiency and longevity, making them a highly attractive option for large-scale water splitting applications. Despite their advantages, several challenges remain, particularly in terms of scalability and cost-effective fabrication techniques. The development of novel material combinations, advanced deposition methods, and optimized device architectures are critical to overcoming these barriers. Researchers are increasingly focusing on understanding the interplay between the various material properties to ensure that the PEC performance is maximized. Furthermore, there is a growing interest in reducing the complexity of the fabrication process while maintaining high performance, which would make mixed metal oxide bilayers more commercially viable.

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