Fabrication of Thin Films by Silar Process: A Review

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ABSTRACT

Thin films have gained significant attraction because of their outstanding unique property for device applications. Thin film deposition can be done using a variety of methods, including chemical bath deposition, chemical vapor deposition, dip coating, sputtering, etc. The present article reveals the Successive Ionic Layer Adsorption and Reaction (SILAR) deposition in detail. This method has the ability to regulate the morphology and optoelectrical properties of the films, its numerous benefits and drawbacks were explored. According to the experimental findings, the films have a lot of promise for use in optoelectronic applications, such as coatings and window materials for solar cells. This review paper aims to present up-to-date knowledge on the synthesis, characterization, and applications of thin films deposited by the SILAR method in light of their current interest.

Keywords: *Thin Films, SILAR and Applications*

1. Introduction:

The study of matter's physical properties has advanced so much over the past century that physics is now separated into a huge number of specialized fields that are frequently quite different from one another. The physics of thin films is a recently created separate and significant field. This article discusses ways for analyzing systems that may differ in terms of all other physical characteristics but only have the commonality that one of their dimensions is extremely small [1]. When the two surfaces are so close to one another, they can have a significant impact on the substance's physical properties and processes, which differ significantly from those of bulk materials. By interacting with one another and reducing the space between the surfaces, new phenomena may emerge [2]. These are some of the reasons thin films have caught the interest of physicists, led to the creation of a separate branch of physics devoted to them, and led to the development of associated technical branches. Since the turn of the century, researchers have been examining the electric characteristics of thin films, including their conductivity, superconductivity, and electron emission [3]. Recent years have seen incredibly quick advancements in this field of study. Research in the field of thin films is still active and undergoing rapid change. Atom-by-atom solids are formed as a result of this process. Despite the enormous complexity of the interaction mechanisms involved in film development, there is a lot of room for creating new, more usable materials [4]. Large area arrays, solar selective coatings, solar cells, photoconductors, sensors, antireflection coatings,

interference devices, polarizers, narrow band filters, IR detectors, waveguide coatings, temperature control of satellites, photothermal solar coatings, etc. are all applications for thin films that are of particular interest. There are many ways to create thin films. Physical and chemical procedures can be distinguished from one another. In physical approaches, some kind of energy is used to transfer the film material from a target source to the substrate [5].

The successive ionic layer adsorption and reaction (SILAR) approach has been used for the past three decades to a method for depositing a range of complex compounds in thin film form that has recently appeared [6]. various methods are used for deposition include vacuum evaporation , sputtering, chemical vapor deposition , electrodeposition process , pulsed laser deposition , spray pyrolysis , sol–gel process , and chemical bath deposition (CBD), successive ionic layer adsorption and reaction (SILAR). The SILAR approach is affordable, straightforward, and practical for large-area deposition. Since the deposition is performed at or close to room temperature, insulators, semiconductors, metals, and substrates that are sensitive to temperature (like polyester) can all be employed [7]. The successive ionic layer adsorption and reaction (SILAR) method, often known as a modified variant of chemical bath deposition, is one of the most recent solutions for the deposition of thin films. It is possible to perform it in glass beakers. The initial supplies are readily available and affordable [8]. Since it's a chemical process, a wide range of substrate types can be coated. By performing the deposition at or near room temperature, metallic substrates are protected from oxidation and corrosion [9]. Deposit in stoichiometry is simple to obtain. Since ions rather than atoms serve as the fundamental building blocks, it is simple to regulate the preparative conditions, resulting in enhanced grain orientation [10].

2. Fundamentals and Theoretical Background of SILAR:

The SILAR method is based on the adsorption of a layer of ionic species onto a surface, followed by a reaction started by the subsequent adsorption of a different ionic species, as the acronym suggests [11]. The result of this reaction is the creation of an insoluble substance, which makes up the thin-film coating. To raise the thickness of the deposited layer, the operation is then repeated [12]. A SILAR setup uses a sequence of phases that are shown in Figure 1 in order to properly manage the deposition process.

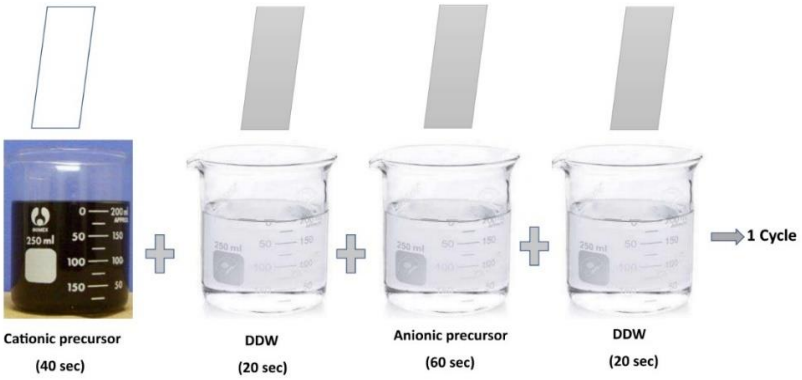

Fig. 1. Schematic representation of SILAR

Rinsing solutions (DDW) are optional, although they are used to get rid of extra material from the substrate after each precursor dip and make sure there is only one ionic layer left before the substrate is submerged in the next solution. We can anticipate faster but less manageable development without rinsing treatments. Without this phase of rinsing, uncontrolled material deposition may occur [13]. Furthermore, if too much precursor is trapped on the substrate's surface, it may be released into the solution containing the second precursor, resulting in the precipitation of the target molecule in the precursor solution [14].

3. Preparation of thin films:

Mahima Ranjan Das et al reported a straightforward, inexpensive, and environmentally friendly SILAR approach was used to successfully synthesize thin films containing NiO nanoparticles on glass and steel substrates. A supercapacitor electrode was made directly from a film on a steel substrate [15]. The zincite (hexagonal) structure of ZnO thin films produced by the chemical deposition method SILAR (successive ion layer adsorption and reaction) are also photoconductive and transparent in the visible and infrared regions (90% optical transmittance) was reported by A. Jim~nez-Gonzfilez et al [16]. The method of fabrication applies successive ionic layer adsorption and reaction (SILAR) to deposit SnS thin films onto uncoated and ITO-coated glass substrates [17]. On glass substrates, thin films of CuS, Cu0.6Zn0.4S, and ZnS were produced using the SILAR technique at room temperature. Investigations have been done into how the annealing temperature affects the crystal structure and optical band gap of thin films. For thin films, a 3-minute annealing period has been determined to be ideal. After being annealed at 400°C, the films have degraded. As a result, we can say that the SILAR method is a suitable method to deposit CuS, $Cu0_6Zn0_4S$, and ZnS thin films which was reported by M. Ali Yildirim et al [18]. N.G. Deshpande et al reported Tin

disulfide (SnS_2) thin films have been created using the low-cost SILAR (successive ionic layer adsorption and reaction) approach. To produce high-quality films, deposition parameters including SILAR cycles (60), immersion duration (20 s), rinsing time (10 s), and deposition temperature (27˚C) were optimized. To investigate the structural, optical, and electrical properties, physical experiments were conducted [19]. Abhishek P. et al. stated that chemically synthesized NiO thin films produced by the SILAR Method were found to be amorphous in character. After being subjected to heat treatment up to 500˚C, the amorphous character remains unaltered [20].

A low-cost method was used to successfully create NiO thin films by altering the deposition period at 5, 10, 15, and 20 min. and non-evacuated chemical spray pyrolysis method at 450 °C. M.M. Gomaa et al. revealed that films were examined and used to create an effective gas sensor for NO_2 detection [21]. The development of layer-by-layer assembly of reduced graphene oxide (RGO) and MnO_2 (MnO_2-RGO SILAR) on a stainless steel current collector, for designing light-weight and small-size supercapacitor electrode, is demonstrated by Milan Jana et al. using the simple, affordable, and additive-free successive ionic layer adsorption and reaction (SILAR) technique [22]. Cu_2O thin films were created using the SILAR (successive ionic layer adsorption and reaction) procedure, according to Soumya Chatterjee et al. They completely characterized the thin films produced using a non-vacuum method and developed them in ambient conditions. CuO thin films have been formed as a result of post-deposition annealing in an oxygen atmosphere [23]. It was reported by Tapio Kanniainen et al. that lead sulfide thin films were produced at room temperature using the sequential ionic layer adsorption and reaction (SILAR) approach on soda lime glass, IT0 and A1203 coated glass, SO_2, (100) Si, and (11l) Si substrates. SILAR uses aqueous precursor solutions to sequentially treat the substrate. Triethanolamine (TEA) was also added to the lead precursor solution as a complexing agent, with a Pb: TEA mole ratio of 1:1. With 0.4 mol dm-3 thioacetamide solution and 0.2 mol dmP3 lead on glass, the growth rate was 0.12 nm each cycle. The films have a metallic aspect [24]. The traditional procedure of depositing Cadmium Sulphate (CdS) and Copper Zinc Tin Sulphate (CZTS) results in a Superstrate and Substrate configuration of a solar cell. The Successive Ionic Layer Adsorption and Reaction (SILAR) approach, which was disclosed by Kaza Jasmitha et al., is modified to achieve the proposed method. For superstrate configuration, CdS thin film is coated on CdS deposited on FTO (Fluorine-doped Tin Oxide) substrate, while for substrate configuration, CdS thin film is coated on CZTS deposited on Mo (Molybdenum)/SLG (Soda Lime Glass) [25]. At room temperature and normal pressure, thin films of zinc peroxide, ZnO, were produced using the sequential ionic layer adsorption and

reaction (SILAR) method. Seppo Lindroos et al. stated that the thin films were formed on glass, quartz, silicon, poly(vinyl chloride), and polycarbonate substrates. Diluted aqueous solutions of ZnCl complexed with H O for the anion and ethylenediamine for the cation H_2O_2 constituents of the film were utilized as the precursors for ZnO films. By annealing in air or in a vacuum, the zinc peroxide film could be converted to zinc oxide[26]. According to Ho Soonmin et al., two alternative deposition processes, spin coating and sequential ionic layer adsorption and reaction (SILAR), were employed to create nanostructured thin films. The merits and disadvantages of various strategies are briefly reviewed in this article. Finally, they review some experimental results in accordance with the literature review [27]. According to a paper by A.T. Ravichandran et al., successive ionic layers by adsorption and reaction (SILAR) were used to build copper oxide thin films onto a glass substrate over the course of 30 cycles [28].

According to J. Puiso et al., lead sulfide or PbS, thin films could be formed using the SILAR method on Si(100) and Si(111) substrates, and the shape and crystallinity of the films could be adjusted by altering the lead precursor. On both silicon substrates, PbS extremely thin films utilizing Pb(Ac)2 as a precursor were very well w200x oriented. The PbS extremely thin films had minor carbon and oxygen impurities but were stoichiometric [29].

According to M.P. Suryawanshi et al., the SILAR process is used to synthesize Cu_2ZnSnS_4 (CZTS) by sulfurizing layers of stacked sulfide precursors. Cu_2SnS_3 and ZnS are stacked in different orders, and this has an impact. It is discovered that the CZTS thin films' PEC performance is significantly impacted by the stacking order of the precursor thin films, which has a significant impact on the CZTS thin films' characteristics. For a stacking order of Mo/ZnS/Cu_2SnS_3, the structural analyses demonstrated the creation of a notable kesterite CZTS phase, and for a stacking order of Mo/Cu_2SnS_3/ZnS, they demonstrated the formation of a secondary Cu_2-XS phase in addition to the prominent CZTS phase. The PEC device with the highest Jsc of 11.68 mA/cm^2, Voc of 0.42 V, FF of 0.37, and power conversion efficiency of 1.81% was made utilizing the sample with the stacking order of Mo/ZnS/Cu_2SnS_3 [30].

Dongzhi Zhang et al. reported on a high-performance liquefied petroleum gas (LPG) sensor based on a layer-by-layer (LbL) self-assembly and successive ionic layer adsorption and reaction (SILAR) technique-fabricated zinc oxide/polypyrrole/lead sulfide quantum dots (ZnO/PPy/PbS QDs) nanocomposite film. The as-prepared ZnO/PPy/PbS QDs sample was examined using the following techniques: FT-IR, XPS, TEM, SEM, EDS, and XRD. In comparison to the ZnO/PPy film sensor, the ZnO/PPy/PbS QDs film sensor showed higher responsiveness, acceptable repeatability, and better selectivity for LPG sensing. The findings

show that the ZnO/PPy/PbS QDs film is a potential raw material for making LPG gas sensors [31] . Tuba Ayr Taşdemirci reported that nickel oxide (NiO) thin films were produced on glass substrates using the Successive Ionic Layer Adsorption and Reaction (SILAR) process at room temperature. Analysis was done on how the annealing temperature affected the structural, morphological, molecular, and optical properties. X-ray diffraction (XRD), scanning electron microscopy (SEM), atomic force microscopy (AFM), Fourier transform infrared spectrophotometer (FTIR), and UV-vis spectrophotometer were each used to analyze NiO thin films [32]. According to Yunus Akaltun et al., the Successive Ionic Layer Adsorption and Reaction (SILAR) approach was used to synthesize NiO thin films on glass substrates at room temperature. Investigations were done into how NiO thin films' structural, morphological, optical, and electrical properties are affected by the film's thickness [33]. Mahima Ranjan Das et al. stated that the successive ionic layer absorption and reaction (SILAR) technology, a straightforward, affordable, and low-temperature wet chemical procedure, has effectively produced NiO thin films [34]. According to Vithoba L. Patil Sharadrao A et al., nanostructured thin films of ZnO were created using the quick and affordable sequential ion layer adsorption and reaction (SILAR) method. Investigations were conducted into how SILAR cycles affected the structural, optical, surface morphological, and electrical characteristics of nanostructured ZnO thin films. The physical and chemical characteristics of the synthesized films were investigated using characterization techniques as XRD, UV-Vis, PL, FESEM, and Hall measurement [35]. Metal chalcogenide thin films were deposited by H M PATHAN and C D LOKHANDE using the sequential ionic layer adsorption and reaction (SILAR) technique. He discussed how to develop metal (binary, ternary, composite, etc.) chalcogenide thin films using the successive ionic layer adsorption and reaction (SILAR) method. Conclusions: (i) Metal chalcogenide thin films can be prepared using the SILAR method; (ii) SILAR is a cheap method, making the process practical for commercial application; (iii) material waste is avoided because no precipitate is formed during this process; (iv) the materials are stoichiometric; and (v) semiconductor doping, desired type for device formation, etc., can be accomplished [36].

4. Structural Analysis:

The film's crystallinity rises with each deposition cycle up to 40, after which it begins to trend in the other direction [15]. ZnO's as-prepared stoichiometry and crystal structure, as well as its optical and electrical characteristics, are considerably altered by heat treatments. The gas environment used during the treatment affects the dark conductivity of ZnO thin films [16]. SnS was identified as the predominant phase in the film by XRD. The corresponding strain and

crystallite size was calculated from the FWHM of XRD spectra [17]. The hexagonal crystal structure of the formed SnS2 thin films is demonstrated by X-ray diffraction (XRD) patterns [19].A strong (200) orientation of the films was discovered by X-ray diffraction examinations [24]. To examine further impurities and perfect the crystalline structure of CZTS and CdS orientation, grazing incidence X-ray diffraction (GIXRD) is utilized [25]. According to X-ray diffraction (XRD), the nucleation process of the precursor solution (film immersion) increases as the crystallinity of the films does. The spherical grains gradually vanish and transform into nanorods with the film's dendrite structure. This is explained by the fact that the film becomes more crystalline with longer immersion times [28].

5. Morphological Analysis:

Surface morphology analyses demonstrated the formation of a highly porous nanoflakes-like network, with the 40-cycle deposited film exhibiting the highest levels of porosity. Additionally, a 40-cycle dipping NiO electrode exhibits the highest specific energies (64.38 Wh Kg^{-1}) and powers (2305 W Kg^{-1}) due to its ability to exploit quick electron transport and readily access electrolyte ions thanks to its porous nanostructure [15]. Nanometer-sized spherical grains were evenly scattered on the substrate's surface, as seen in SEM pictures [17]. SEM pictures of sprayed NiO layers at 5 and 10 min of deposition time reveal a porous structure with a randomly aligned honeycomb topology [21]. The MnO_2-RGO SILAR contains RGO and MnO_2 in a homogeneous distribution, as shown by pictures obtained using transmission and field emission scanning electron microscopy. Comparing the LbL (MnO_2- RGO SILAR) to the hydrothermally produced MnO_2- RGO (MnO_2- RGO Hydro), the latter exhibits superior physical and electrochemical properties [22]. The films seemed relatively rough and contained grains whose diameters roughly matched the film's thickness, according to photographs from a scanning electron microscope (SEM) [24]. Images taken using scanning electron microscopy showed that the films were homogeneous and uniform [26]. Studies using scanning electron microscopy (SEM) and X-ray diffraction (XRD) revealed that all of the films exhibit polycrystalline structures and are well covered by glass substrates. With increasing film thickness, the crystalline and surface characteristics of the films improved [33]. XRD and FESEM were used to evaluate how the deposition cycle affected structural and morphological properties. An analysis of the surface's morphology reveals the development of a highly porous network, which offers more active sites and a path for the deposition of electrolyte ions [34].

6. U-V Studies and Thickness Measurement:

As-prepared samples (of thickness 667) exhibit a dark conductivity of 1.50 X 10^{-6} [fl-cm]-I, whereas a maximum dark conductivity of 2.70 X 10^{-2} [fl-cm]-I was attained after

successive heat treatments in 0_2 and H_2 at 350°C. After annealing, the optical bandgap of the as-prepared ZnO, 3.38 eV, drops by 0.125 eV [16]. The optical direct bandgap was calculated from UV-Vis spectrophotometry to be 1.43 eV. Near band-edge emission and higher energy deep-level transitions were seen in photoluminescence reported by Biswajit Ghosh et al [17]. UV-Vis-NIR Spectrophotometer is used to conduct individual optical research on CdS and CZTS. A 3A class solar simulator was used to assess the current-voltage (J V) investigations for Voc, Jsc, and fill factor under a single sun's illumination (1 0 0mW cm/2) [25].UV spectroscopy was used to characterize the films as well [26].

7. EDX and AFM Studies:

Tin disulfide (SnS (2.02)) was found to have elemental ratios that were similar to those found in energy dispersive X-ray analysis (EDAX). Atomic force microscopy (AFM) revealed that the film is homogeneous and that the substrate surface is completely covered with tiny, overlapping spherical grains. 2.22 eV of direct band gap was attained[19]. AFM surface morphology investigations show that heat treatment causes the surface of the films to become smoother, as seen in the 2-D AFM images. According to estimates, annealed films have a surface roughness of roughly 320nm and unannealed samples have a roughness of 1.11m. The optical transmittance in the visible region of the electromagnetic spectrum for the annealed films is found to be higher than for the unannealed sample [20].

8. Applications:

Because of this, the deposited NiO thin films' total capacitance performance makes it a potential contender for an advanced, highly effective energy storage system [15]. Measurements of photosensitivity have been conducted, and it has been discovered that while photosensitivity increases with light intensity, it decreases with increasing annealing temperature. These films are extremely light-sensitive. This is one of the first studies that led to the deposition of the CuZnS thin films by using the SILAR method and light effect (under 150, 300, and 500W cm^2 light emission) on I-V measurements of CuS and CuZnS thin films. As a result, we can say that the SILAR method is a suitable method to deposit CuS, Cu0.6Zn0.4S, and ZnS thin films which was reported by M. Ali Yildirim et al [18]. Two distinct peaks of photoluminescence (PL), corresponding to red and green emission, were visible. Ag/SnS2 junction had I-V characteristics akin to Schottky diodes. The predicted barrier height was 0.22 eV. Tin disulfide shows n-type conductivity, according to thermoelectric power (TEP) parameters [19]. Nitrogen dioxide (NO_2) detection performance was tested at various operating temperatures and NO_2 concentrations. It was discovered that the sprayed NiO thin film exhibits good selectivity and stability with a maximum sensitivity of 57.3% for 20

ppm NO_2 at 200°C after a 5-minute deposition time [21]. MnO_2-RGO SILAR is used as the positive electrode in an asymmetric supercapacitor device, while thermally reduced GO (TRGO) is used as the negative electrode. The developed cell displays a high power density of 23,200 W kg1, a high energy density of 88 Wh kg1, and 79% capacitance retention after 10,000 charge-discharge cycles [22]. With the help of scanning tunneling spectroscopy (STS) measurements, we were able to pinpoint the materials' band boundaries in relation to their Fermi energies. The heterojunction devices made of NiO, Cu_2O, ZnO, and SnO_2 had an energy level that resembled a staircase. This allowed for easy passage of electrons and holes to the opposing electrodes, operating as all-oxide thin-film solar cells with an energy conversion efficiency of more than 1% [23]. The films Cu-O vibrated in the range of 484 cm^{-1}, 499 cm^{-1}, and 511 cm^{-1}, according to FTIR results and confirmation [28]. The values of the energy band gap as a function of the film thickness were used to compute the values of the refractive index (n), optical static constant (eo), and high-frequency dielectric constant (e1). At normal temperatures, the films' resistivity ranged from 4.1 to 802.1 X cm as film thickness increased [33]. After 1000 cycles, the NiO electrode displayed long-term cycle stability with 90% capacitance retention. The produced electrode's appealing electrochemical performance is ideal for the production of high-quality supercapacitors for industrial use [34]. The gas sensing performance of the nanostructured ZnO thin films synthesized at 30 SILAR cycles is improved, and they show much greater responses (5% per ppm). The innovative nature of this study is demonstrated by the sensitive gas sensor employed to detect tiny levels of NO_2 [35].

Table 1. Experimental parameters of selected SILAR-deposited binary metal oxides.

Cationic precursor	Anionic precursor	Number of Cycles	Rinsing	Annealing [°C]	Dipping time (cationic, anionic)	Comment/outcomes
CuO						
0.05–0.15 m $CuSO_4$ + NH_3	Water, 90 °C	–	No	–	15 s, 20 s	Optical properties are affected by precursor concentration [5].
0.1 m $CuSO_4$	0.1 m NaOH	–	water	400 °C	20 s each	Nanoflower-type morphology [6].
0.05 m $CuSO_4$	0.02 m NaOH, 45–85 °C	80	water	–	10 s each	Morphology is dependent on precursor temperature [9].

0.1 m $CuSO_4$ + NH_3	Water, 80 °C	10, 20, 30, 40.	Water only at the end	–	10 s,15 s	Grain size is dependent on number of cycles [13].
0.1 m $Na_2S_2O_3$ 0.1 m $CuSO_4$	0.1 m NaOH, 70 °C	90, 100, 110120.	No	350 °C	5 s each	Thickness and grain size are dependent on the number of cycles [17].
Cu_2O						
0.1 m $Na_2S_2O_3$ 0.1 m $CuSO_4$	0.2 m NaOH, 40–80 °C	–	No	–	10 s each	Crystal quality and density of nanowires are dependent on bath temperature [12].
1 m $Na_2S_2O_3$ 1 m $CuSO_4$	1 m NaOH, 70 °C	10, 15, 20, 25,30.	No		5 s each	Bandgap is dependent on cycle number [14].
ZnO						
0.01 m Zn $(NO_3)_2$	0.02 m $N_2H_4{\cdot}H_2O$, 75 °C	60, 120, 180	water	350 °C	10 s each	Crystal orientation is dependent on cycle number [15].
0.05–0.15 m $[Zn(NH_3)_4]^{2+}$	1% H_2O_2, 80 °C	50, 100, 150,200	water	400 °C	20 s, 5 s	Nanorod morphology. Diameter and thickness related to Zn concentration [16].
0.025 m Zn $(CH_3CO_2)_2$ + NH_3	1% H_2O_2	10, 20, 30, 40	water	300 °C	25 s, 30 s	Particle size is related to cycle number [18].
0.1 m $ZnSO_4$ + NH_3	Water, ≈100 °C	100	No	200 °C	2 s each	Crystal size and orientation are dependent on the type of substrate [19].

9. Conclusion and Future Outlooks

Thin films play a significant role in numerous technologies, and the processes and procedures used to create them are themselves an expansive area of scientific inquiry and

technical advancement. There are two categories in this area: both physical and chemical depositions. The current review is concentrated on a particular chemical technique called SILAR, or Successive Ionic Layer Adsorption and Reaction, which has attracted attention recently due to the benefits it provides over other chemical techniques. The advantages of atomic layer deposition are combined.

SILAR is appropriate for the deposition of complex multicomponent nanostructures with advanced functions as well as thin films of oxides, peroxides, hydroxides, sulfides, tellurides, and selenides. In this review, emphasis is placed on oxide systems and their applications as well as the impact of experimental parameters on the composition and characteristics of deposited films. More specifically, the use of oxide films created by SILAR for solar cells, photoelectrochemical water splitting, and energy storage devices (supercapacitors) is detailed in depth. For a device to be engineered precisely and intelligently, a thorough grasp of the links between processing, structure, characteristics, and performances is essential. One goal of a review is to gather sufficient data from the literature and attempt to correlate it in order to provide the clearest, most complete picture of these correlations. This review makes an attempt to do so, and while it can provide a persuasive scientific analysis for some elements, there are many others that require more in-depth research.

We think that this work will help to shape future research on this technique, which is extremely intriguing and encouraging for the straightforward, affordable production of nanomaterials of excellent quality and with highly controllable functional characteristics.

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