

Chemoresistive Metal Oxide-Based Sensors Synthesized Through Physical Vapor Deposition Techniques for Gas Detection

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Abstract

In our day-to-day lives, we are regularly exposed to a wide spectrum of dangerous gases. Their origins vary, ranging from industrial activities to objects found within our very homes. Naturally, there is an interest in developing cost-efficient and durable devices that can successfully track these gases within our environment. One such candidate is represented by chemoresistive gas sensors based on metal oxides. This is due to their simple architecture and the possibility of scaling down their size, making them valid contenders for future advancements in portable gas sensors. This review focuses on chemoresistive gas sensors that have been obtained through different Physical Vapor Deposition (PVD) methods, which are easily scalable for potential technological transfer towards commercialization or are already exploited at the industrial level, and how varying different deposition parameters impacts the structure of the active material, thus modifying the gas sensing properties of the device. In this review, we report results obtained for different metal oxides: WO₃, ZnO, CeO₂, TiO₂, NiO, and SnO₂. The main findings of these studies revealed that the sensor's response was highly impacted by oxygen deficiencies within the deposited material, the specific surface area, and the thickness of the film. Moreover, this study also delves into different strategies of functionalization that result in improved gas sensing properties. Thus, we herein report how tailoring functional properties modifies the gas sensing performance of different metal oxides.

Keywords: gas sensor; chemoresistive; metal oxide; PVD deposition; thin films

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1. Introduction

Rapid industrialization has led to a significant increase in the concentration of pollutants released into the atmosphere, exposing humans to a multitude of harmful gases and thus affecting our day-to-day lives [1]. The combustion of fossil fuels in the oil, gas, and automotive industries is the primary source of these emissions, releasing greenhouse gases like methane (CH₄) and carbon dioxide (CO₂) [2–4]. This has resulted in a rise in average temperatures on a worldwide scale and an increase in the frequency of extreme

weather events, such as tropical nights, in regions where such phenomena were not previously recorded [5,6]. Moreover, we are also exposed to other toxic compounds in indoor spaces. Volatile organic compounds (VOCs), such as formaldehyde (CH_2O), which is a highly toxic and volatile compound, are commonly found in homes [7,8]. A study has shown that the levels of indoor pollution are affected by the presence of this compound, as it is used in the manufacturing of construction materials and furniture [9,10]. This constant risk of exposure to a multitude of gases, both indoors and outdoors, underlines the need for continuous monitoring of our environment.

As highlighted in a comprehensive review by Ritu Malik et al., a clear trend emerges regarding the preferred deposition techniques for synthesizing nanomaterials for gas sensing. That is, as they are being used far more frequently than PVD techniques, synthesis methods, including sol-gel, hydrothermal, electrospinning, and Chemical Vapor Deposition, are the conventional production methods [11,12]. As they use different physical and chemical principles, discussions are held on their advantages and disadvantages regarding their usage in the development of gas sensing devices. Employing the CVD method allows the precise control of surface morphology and offers tunable deposition rates coupled with reasonable costs for the processing of materials [13,14]. Although it presents these benefits, there are serious hazardous risks due to the high temperature needed to process the materials and the usage of highly toxic precursors [15]. Electrospinning is an extremely versatile, cost-effective, and relatively simple-to-employ synthesis method. The current issues underlying electrospinning deposition are poor reproducibility and scalability [16–18]. Moving on to the hydrothermal synthesis approach, its advantages include an easy-to-understand process, the ability to modify the microstructure of the deposited material, and the fact that it is a safe and environmentally friendly way to create active materials for gas sensing [19,20]. The challenges once again arise when scaling up the process due to inconsistencies in the resulting material [21]. Sol-gel synthesis offers precise control over the chemical composition of the resulting product, presents low-temperature deposition possibilities, and a wide range of materials can be deposited using this method [19,22]. However, as with the other three previously discussed techniques, the main challenge shared by these synthesis methods is their limited usage in industrial manufacturing [22].

Inherently, as one of the main goals when producing sensing devices is to lower costs and speed up production, there is a need for alternative deposition methods that are scalable to large areas. Although the active materials deposited through the aforementioned conventional methods present increased porosity, different PVD methods showcase the possibility of increasing sensor production through batch processing. In a recent study, Yu Guo et al. successfully developed a batch of Pd/WO_3 -based sensors during deposition of the same electron beam, showing similar performance across all seven tested devices. They showcased that this PVD method presents high potential for future industrial manufacturing of such technology [23]. Another deposition method that offers high promise in this regard is magnetron sputtering. In a review, Pedro Catalão Moura et al. assessed the advantages of this synthesis method for sensor fabrication. The main takeaways that the authors provided were the scalable nature of this method and great control over the morphology and uniformity of the active material over large-scale areas, thus presenting high promise for batch production [24]. In Figure 1, the advantages of pulsed laser deposition (PLD), magnetron sputtering (MS), and electron beam deposition are presented.

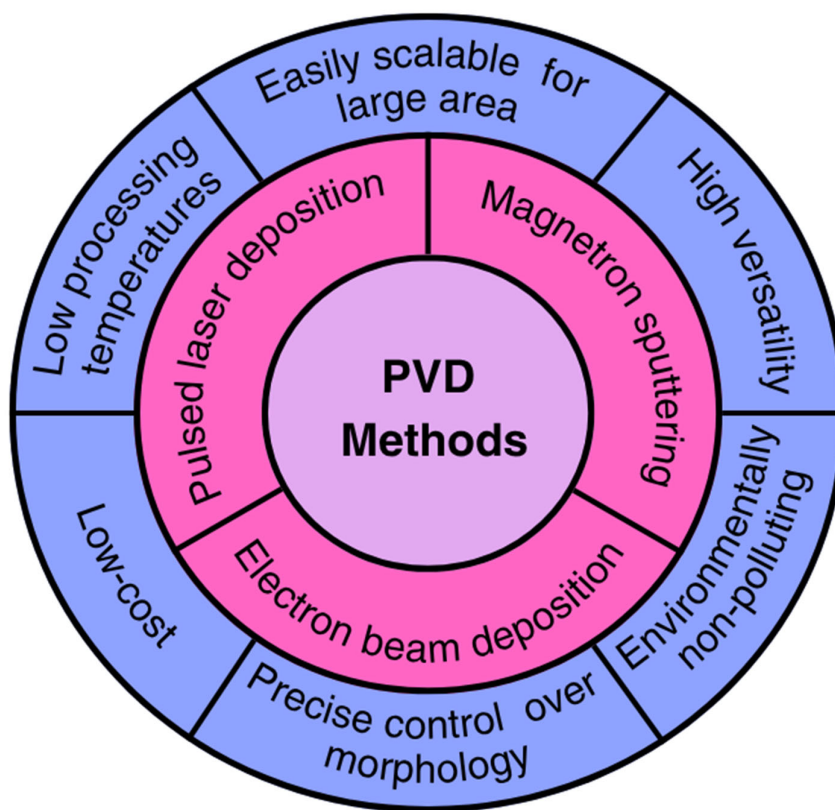


Figure 1. Diagram showcasing the advantages of different PVD methods used in the production of chemoresistive gas sensing applications.

Many researchers have tackled the subject of metal oxide-based chemoresistive gas sensors, as they present high potential in the field. In a review, Shuai Ma et al. [25] elaborate on the subject of nanoscale heterojunctions based on metal oxides. In their work, they mainly focus on how these heterojunctions enhance the sensor's properties. They mostly include materials obtained through conventional fabrication methods. In another review, R. Xavier and K. Sivaperuman et al. [26] survey the literature in a brief study of semiconductor MO gas sensors obtained through PVD synthesis techniques. Their study presents a useful compilation of devices that have mostly been obtained through magnetron sputtering. Contributing to the field of MO-based chemosensors, Abderrahim Moumen et al. [27] offer an extensive view on p-type metal oxide-based devices obtained through a wide variety of deposition methods. Our review offers a unique viewpoint on recent developments in this area of study. We offer a compilation of specific MOs and deposition methods that, in combination, present high potential in developing the industrial production of chemoresistive-type sensors.

This paper focuses on recent advancements made in the field of chemoresistive gas sensors, where metal oxides are often used as the active material, as they present reliable interactions with target gases and low cost of fabrication [28]. Thus, we provide a comprehensive analysis of several materials of importance within this category, specifically focusing on CeO_2 , ZnO (and its dopants), NiO , TiO_2 , WO_3 , and SnO_2 oxide materials. The performance of these materials was evaluated based on parameters that define a gas sensor's efficacy: operating temperature, sensor response, response and recovery times, limit of detection, and the specific gas concentration to which the device was exposed. By examining these parameters, we aim to provide a detailed overview of the current state of the art for these materials in sensing applications. This article is

dedicated to sensors in which the active material is a thin film obtained through Physical Vapor Deposition (PVD) techniques, as we look forward to analyzing materials fabricated via pulsed laser deposition (PLD), magnetron sputtering (MS), and electron beam evaporation (EBE). Importantly, this review briefly explores functionalization methods utilized recently in order to improve gas sensing properties of metal oxides. Moreover, we focus on results obtained solely on metal oxide-based chemoresistive gas sensors thanks to recent advancements in upscaling PVD synthesis methods.

2. Deposition Methods

Material synthesis methods are broadly divided into two categories: Physical Vapor Deposition (PVD) [29] and Chemical Vapor Deposition (CVD) [30]. Depending on the application, researchers alternate between the two methods, as they present different weaknesses and strengths. For example, CVD is preferred for applications involving complex material geometries, as the deposition process uniformly coats the entire surface of the substrate. In contrast, PVD is a “line-of-sight” synthesis method, which leads to incomplete coverage and shadowing problems on complex surfaces [31]. On the other hand, PVD methods are considered more environmentally friendly as they typically avoid the usage of toxic and reactive gases and precursors, which are employed in CVD to achieve chemical reactions [32].

Pulsed laser deposition (PLD), electron beam evaporation (EBE), and magnetron sputtering (MS) are all PVD methods [33]. These thin film deposition techniques involve transforming a solid source material into the vapor phase through different physical processes. The vapor then condenses on the surface of a desired substrate, forming a solid thin film. To prevent the incorporation of impurities and ensure the high quality of the deposited film, these synthesis methods require vacuum chambers [34].

These PVD methods have different working principles, and by default, they present different experimental setups. This leads to differences in cost-related aspects, an important subject when developing materials for industrial-scale use. At the same time, the different physical processes used in these deposition methods cause some of them to deposit certain materials more effectively than others. Thus, this section will focus on a comparative study of the advantages and disadvantages of these PVD synthesis techniques.

2.1. Pulsed Laser Deposition (PLD)

The pulsed laser deposition (PLD) method is a PVD technique that uses a high-power, nanosecond-pulsed laser whose beam is focused onto a solid target to ablate a material and deposit it onto a substrate [35–37]. The rapid, localized heating by laser pulses generates a plasma plume containing energetic ions, atoms, and molecular species [36]. The plume is directed towards the substrate thanks to the geometry of the setup, as the target is placed parallel to the substrate [38].

One of the major reasons why the PLD technique is at times the preferred deposition method over other PVD methods is its unique property, known as stoichiometric transfer [39], meaning that the elemental composition of the deposited film accurately matches that of the target material [40]. Thus, the PLD method is favored when it comes to synthesizing materials with complex structures, like metal oxides [37,41,42] and perovskites [38].

Common issues with the pulsed laser deposition (PLD) method are problems with film uniformity and the presence of so-called particulates [35]. The laser ablation process can lead to the formation and ejection of macroscopic droplets from the target, affecting thin film uniformity [40]. This problem is mitigated in advanced PLD systems, particularly those designed for large-area deposition. The component responsible for ensuring the

uniformity of the obtained thin films is called a “droplet trap,” which stops macroscopic particulates from being deposited on the film’s surface [43]. Another aspect that negatively affects the uniformity of PLD-deposited thin films is the dynamic nature of the plasma plume. When varying the gas pressure during deposition, plasma plumes will vary in size [40], a fact that negatively impacts the usage of this method in large-scale industrial applications due to the larger number of calibration depositions required to obtain good uniformity of thin films.

The PLD method is quite complex, as the experimental setup consists of a laser, positioned outside the vacuum chamber, with the beam redirected and introduced into the reaction chamber through different optical components like lenses and mirrors [33]. Moreover, the system requires high-power lasers, a vacuum chamber, and mechanical components, all leading to high initial and running costs [44]. Figure 2 shows the experimental setup of a large-area PLD system [45].

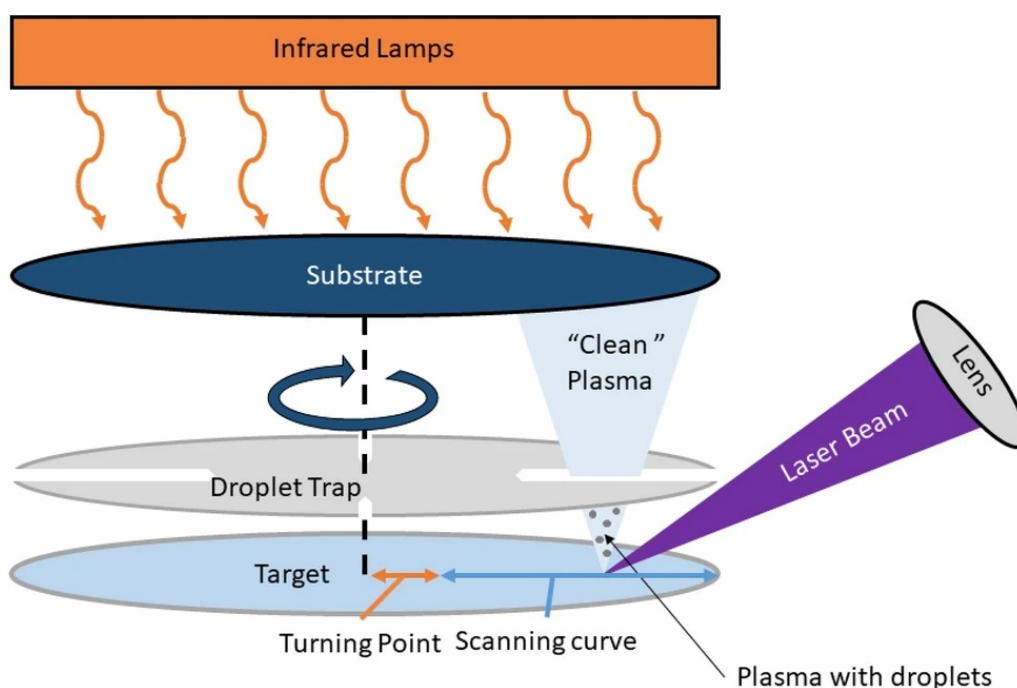


Figure 2. Experimental diagram for the large-area PLD method (reused with permission from [45]).

An important detail to mention is that the PLD method can be conducted in the presence of reactive gases like oxygen and nitrogen. This aspect allows for the control of film stoichiometry by inducing defects, such as oxygen vacancies. Defect engineering is really important when designing chemoresistive gas sensors because, through this action, the material’s gas sensing performance can be tailored and enhanced [46].

Another important factor to consider in the PLD method is the control of substrate temperature and pressure during the experiment, which can affect the thin film’s crystallinity. Depending on the parameters used, different grain sizes are obtained, which, in turn, modify the specific surface area of the active material. This aspect may positively impact the overall performance of a sensor [47,48].

2.2. Magnetron Sputtering

This PVD method utilizes a working gas, typically argon (Ar). A high-voltage potential is applied between the cathode (target) and the anode (placed behind the substrate), which ionizes the argon gas, creating a plasma. The positively charged argon

ions are then accelerated towards the target that is negatively biased, where they collide with it, effectively removing material from the target's surface [49,50].

What differentiates this from other sputtering techniques is the presence of a strong magnetic field behind the target. This field confines the electrons near the target surface, increasing their path length and, in turn, improving their chances of ionizing the working gas. This aspect results in higher deposition rates [50,51].

Magnetron sputtering can be powered by either a DC or RF source, which allows for the deposition of a larger range of materials. DC power is generally used for conductive materials, while radio-frequency (RF) magnetron sputtering is used to deposit materials with a high dielectric constant [49]. In the case of insulating materials, an RF source prevents positive charge buildup on the target surface, which would negatively impact the deposition process by reducing deposition rates [50].

The medium to high deposition rates that magnetron sputtering exhibits make this deposition technique more desirable when compared to other PVD methods. Moreover, substrate temperature can be kept relatively low compared to other deposition methods while still obtaining high-quality, uniform thin films. Thus, thin films obtained through this synthesis method are important because of their usage in applications where structures require low-heat processing to achieve temperature-sensitive characteristics [52].

Magnetron sputtering is a deposition method that offers versatility, as it can deposit a wide range of materials. It has been reported that metals, different oxides and alloys have been deposited using this method [53]. Another advantage of this deposition method is its efficiency in industrial settings, owing to its high repeatability [50]. This means that by using different conditions of pressure, power, and temperature, this deposition method offers high control over the thickness, crystallinity, and structure of deposited thin films. This represents a big benefit when it comes to the large-scale development of 2D structures used in gas sensing applications [54]. In Figure 3, a typical schematic of a magnetron sputtering experiment is showcased [49].

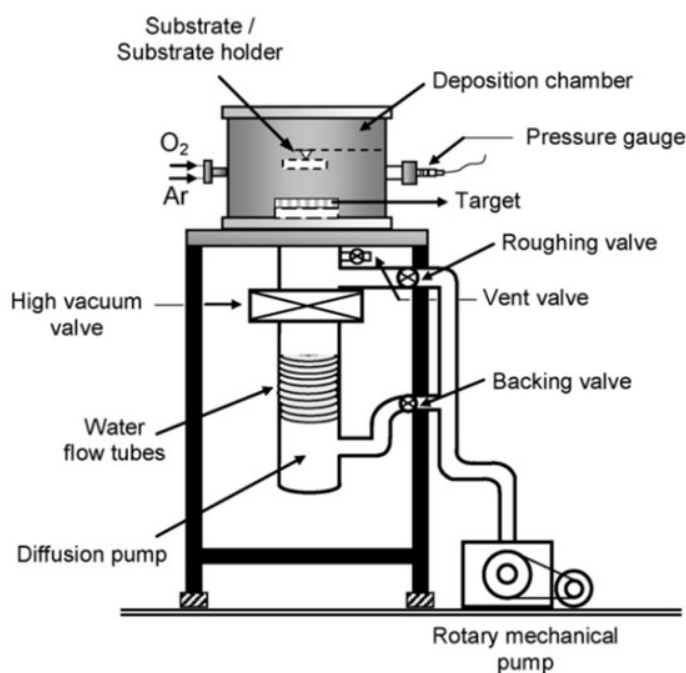


Figure 3. Experimental setup for the magnetron sputtering deposition method (reused under the terms of the Creative Commons CC BY 4.0 license from [49]).

One of the main disadvantages of the magnetron sputtering deposition method is the high initial cost, primarily due to the requirement for a high-performance high-vacuum system. Furthermore, the operational costs are also significant as there is a constant need for high-purity argon gas to sustain the plasma, thus contributing to running expenses. Another cost-related issue is the inefficient use and erosion of sputtering targets. In the magnetron sputtering process, as mentioned before, a magnetic field facilitates the formation of a denser plasma on the surface of the target. Although it provides higher deposition rates, the shape of the plasma leads to uneven erosion of the target's surface, commonly known as a "racetrack." Thus, it is possible that a substantial portion of the target material could remain unused during its use [50,55,56].

2.3. Electron Beam Evaporation (EBE)

Electron beam evaporation is a PVD method that uses an electron gun to heat a material of interest. The source material, which is often found in the form of pellets, is placed in a crucible made of a high-temperature-resistant material to prevent the contamination of the thin film during deposition [57].

The electron gun is usually made out of a tungsten filament, to which a high current is applied, causing thermionic emission of electrons. An accelerating voltage and a magnetic field are used to direct and focus the electron beam onto the source material in the crucible. Depending on the material's melting point, the electron beam will either evaporate or sublime the pellets; then, the vaporized particles travel through the vacuum chamber and condense onto the substrate [57].

The variables that can be controlled during EB-PVD are the power and focus of the electron beam and the substrate temperature, which allow for the precise control of deposition rates and film thickness. The setups are often coupled with monitoring techniques, such as a quartz crystal resonator, which provides real-time feedback on the deposited film thickness [57].

The electron gun in EB-PVD provides intense localized heating. Thus, this deposition method enables the synthesis of materials with high melting points [58,59]. This capability distinguishes it from other deposition techniques, such as pulsed laser deposition (PLD), which is typically used to synthesize complex materials that require stoichiometric transfer. Moreover, a large number of materials can be deposited through EB-PVD, including metals, alloys, and dielectrics, making this deposition technique versatile [58–62].

One of the main challenges that may arise with this deposition method is that the electron beam is focused on a small area within the crucible. This localized heating can cause uneven evaporation of the pellets placed in the hearth, potentially damaging the material holder. As a result, unwanted material deposition on the substrate may happen if this issue is not properly controlled [57].

As with most PVD methods, Electron Beam Physical Vapor Deposition (EB-PVD) is a line-of-sight deposition technique. This means that the vaporized material travels in a straight path from the crucible to the substrate. Thus, problems with uniformity and coverage arise when depositing a material on substrates with complex geometries or surfaces that present shadowed areas. This can result in parts of the substrate remaining uncovered. Therefore, this deposition method is best suited for substrates with simple geometries to ensure the deposition of high-quality, uniform thin films [63]. A standard schematic of an electron beam deposition experiment is presented in Figure 4 [64].

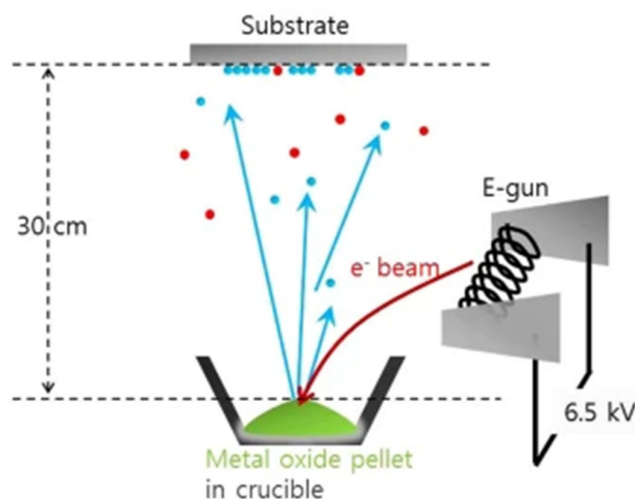


Figure 4. Experimental setup for the electron beam evaporation deposition method (reused under the terms of the Creative Commons CC BY license from [64]).

3. Metal Oxide-Based Chemosensors

3.1. WO_3

Tungsten oxide (WO_3) is a transition metal oxide with a reported bandgap value between 2.6 and 3 eV [65]. When using PVD methods, depending on the deposition conditions, such as substrate temperature and partial pressure, the crystalline structure of the material undergoes phase transitions. It has been observed that WO_3 presents cubic, hexagonal, tetragonal, triclinic, orthorhombic phases [66,67].

An important aspect that needs to be taken into consideration when trying to improve a sensor's overall performance is the role of oxygen vacancies and how their interaction with the gas may lead to improvements in sensing properties [68,69]. For example, when a reducing gas interacts with an n-type semiconductor, the presence of a larger number of oxygen vacancy sites leads to the donation of more electrons to the active material, which then translates into a larger decrease in electrical resistance. This can be done during a deposition experiment by varying the partial pressure of oxygen. In a reactive RF magnetron sputtering experiment, M. Gillet et al. synthesized thin films of WO_3 and varied the partial pressure between depositions. It was observed that as the experiments were done under lower pressures, the number of oxygen vacancies increased and thus the activation energy decreased [66]. Another method to modify the amount of oxygen vacancies in a material is through post-processing experiments, such as thermal annealing [70–72]. The table below compiles the gas sensing results obtained for various WO_3 thin films produced by PVD methods (see Table 1).

Using DC reactive magnetron sputtering, Dwivedi et al. synthesized WO_3 thin films at room temperature under varying oxygen partial pressures to study the effect of oxygen vacancies on gas sensing behavior. During the deposition itself, the values for pressure and power were kept constant at 30 mTorr and 100 W. All samples were post-annealed at 400 °C in air. FE-SEM revealed details of the thin film's topography, showing a porous, rough structure. Moreover, the surface roughness of the active material was determined through AFM, exhibiting an average roughness of ~ 0.17 . XPS analysis showed that films deposited at a higher oxygen flow rate (35 sccm) contained fewer oxygen vacancies compared to those prepared at lower flow rates. When tested toward 50 ppm NO gas, the sample deposited at 35 sccm O_2 exhibited the best sensing performance, with an optimal operating temperature of ~ 275 °C, where the response reached a maximum value of ~ 147 . At this operating temperature, it also presented the best results in terms of response and

recovery times, which were 19 and 17 s, respectively. These findings highlight the crucial role of oxygen stoichiometry in modifying the sensitivity and response of WO₃-based NO gas sensors [73].

Table 1. Gas sensing properties values for WO₃ thin films found in the literature.

Deposition Method	Substrate Temperature	Target Gas	Concentration	Operating Temperature	Response	Response Time (s)	Recovery Time (s)	Ref
RF magnetron sputtering	RT	NO ₂	0.68	RT	2.2	30	21	[72]
DC magnetron sputtering	RT	NO	50	275	147	19	17	[73]
Pulsed laser deposition	RT	NO ₂	20/5	75	93/5	20/20	80/80	[74]
Electron beam evaporation	RT	Alcohol	10	240	30	140	100	[75]
DC magnetron sputtering	800	CH ₄	25	200	8.33	20	25	[76]
RF magnetron sputtering	200	H ₂	100	RT	3.24	1.6	2.8	[77]
DC magnetron sputtering	RT	NO ₂ /NO	50	150/200	196/100	113/39	79/99	[78]
DC magnetron sputtering	-	C ₃ H ₆ O	4	300	-	45	251	[79]
DC magnetron sputtering	RT	NO	100	250	22.7	172	86	[80]
RF magnetron sputtering	300	NO	1	250	25.07	107	45	[81]

In another study, WO₃ thin films were deposited via nanosecond pulsed laser deposition (PLD) using a Nd:YAG laser at 20 J/cm² fluence and 10 Hz repetition rate. The films were grown at room temperature and later annealed at 400 °C for 3 h in air to assess how post-deposition treatment influences the sensing performance of the device. SEM and AFM analyses revealed nanogranular surface features with droplet-like structures characteristic of PLD and a slight increase in surface roughness after annealing. EDS measurements indicated a higher oxygen content in the annealed films, suggesting a reduction in oxygen vacancies compared to the as-deposited ones. Electrical measurements showed that sub-stoichiometric WO₃ films exhibited higher conductivity and stronger temperature dependence, a property that was attributed to their higher concentration of oxygen vacancies. When tested toward NO₂ (5–20 ppm) at an operating temperature of 75 °C, the as-deposited films displayed higher sensitivity, with a maximum response of ~93 at 20 ppm and response/recovery times of 20 s and 80 s, respectively. These results demonstrate that controlled oxygen vacancy engineering strongly affects both the conductivity and gas sensing characteristics of WO₃ thin films [74].

Using electron beam evaporation, E. Amani et al. developed WO₃ sensors for alcohol detection. The thin films of WO₃ were deposited at room temperature using different deposition angles (0°, 30°, 60°), and gas sensing measurements were taken at an operating temperature of 240 °C. The best responses were obtained from the sample deposited using an angle of 30°. Analysis showed that its structure was more porous than the other samples obtained, resulting in a larger effective surface area. Through AFM, it was determined that this sample presented smaller grains and a larger number of grain boundaries, thus generating a higher response by facilitating the penetration of more oxygen molecules into the film surfaces. The sample was tested at different ethanol concentrations, showing a stronger response to the alcohol vapor as the concentration increased. The WO₃ sensor showed a reliable response starting at a 10 ppm concentration, showing a change in resistance of 30% and response and recovery times of 140 and 100 s. A maximum response of 68% was recorded when the sensor was exposed to a concentration of 50 ppm of ethanol [75].

A study focused on the impact of substrate temperature on a film's crystalline structure and how it impacts a sensor's gas sensing capabilities. Santosh Kumar et al. deposited WO₃ thin films using DC magnetron sputtering and varied the substrate

temperature from 200 °C to 800 °C in increments of 200 °C. Through XRD, it was determined that the WO₃ thin films had poor crystallinity, presenting small peaks associated with a monoclinic phase. Increasing the temperature of the substrate resulted in a phase change, as a peak corresponding to hexagonal WO₃ emerged in the XRD diffraction pattern. FE-SEM images revealed the importance of substrate temperature on the structure of the film, as the WO₃ thin film deposited at 800 °C presented grain growth, thus resulting in a less porous surface with a smaller specific surface area. For gas sensing applications, smaller grain sizes facilitated better interaction between the gas molecules and the active material, as they presented more active sites. Even though all other parameters were kept the same during deposition, elemental composition studies revealed that the thin films deposited at higher temperatures presented lower atomic weights of O₂; thus, more oxygen vacancies were present, leading to better responses. The WO₃ sensor's optimal operating temperature was found to be 200 °C, as it exhibited a reliable response of 8.33% to 25 ppm of CH₄. To study its selectivity, the active material was also exposed to CO and H₂, which showed a lower response than the gas of interest. Moreover, when exposed to CH₄, the sensor presented better response and recovery time capabilities. The sensor successfully absorbed the gas molecules in 20 s and returned to its baseline resistance in 25 s [76].

Employing RF magnetron sputtering, Ramya Barathy et al. studied the impact of oxygen vacancies on the H₂ gas sensing mechanism of WO₃ thin films. Three different depositions were performed, where the partial O₂ pressure was varied between 10% and 20% from experiment to experiment. All of the other parameters were kept constant throughout all of the depositions. The thin films were also subjected to annealing at 800 °C under Ar pressure. Through XPS, it was determined that the film deposited at a partial pressure of 10% presented the highest concentration of oxygen vacancies, and the material deposited at 20% pO₂ presented an almost stoichiometric structure. XRD analysis revealed that at a lower O₂ partial pressure, there are changes in the morphological characteristics of the thin film. Through FE-SEM, it was revealed that the films deposited at lower pressure showcased a more porous structure and modulation of the grain boundaries, leading to improved interaction between the surface and the gas of interest. The optimal operating temperature for the WO₃ chemoresistive gas sensor was found at room temperature, as it presented a reliable response to the gas of interest, H₂. Moreover, the sensor was able to respond quickly and recover after being exposed to a concentration of 100 ppm of H₂. The active material successfully absorbed the gas of interest in 1.6 s, yielding a response of 3.24, and managed to desorb the gas molecules in a short time of 2.8 s [77].

3.2. ZnO/AZO

In a study, ZnO thin films were deposited by electron beam evaporation to evaluate gas sensing properties. The depositions were conducted on two different substrates: one was deposited on a silicon (Si) substrate and the other on an alumina (Al₂O₃) substrate. SEM and AFM analyses showed that the film structure varied depending on the substrate used. The active material deposited on alumina exhibited smaller grain sizes in comparison with the structure deposited on Si. The smaller grain size facilitated better gas adsorption at the surface, as such films presented more active sites for the active material to interact with molecules. Thus, further measurements were conducted on the ZnO thin films deposited on the alumina substrate. Gas testing was conducted at room temperature, and the sensor was exposed to different concentrations of O₃, with values ranging from 50 to 1150 ppb. It was revealed that the sensors recorded the highest response of 5.1 to 450 ppb of O₃. The sensor presented an extremely fast response of 2 s and a fast recovery time of 15 s. Further measurements using higher concentrations of gas revealed a reduction in

the sensor's sensitivity. To understand why the sensors' performance deteriorated, topographical studies were done, which revealed that gas exposure led to the formation of micron-scale features on the active material's surface. Through XPS, it was determined that the surface had also undergone changes in terms of chemical composition, as the amount of Zn decreased by 80% compared to the initial recorded values. Thus, at room temperature, the measurements resulted in rapid degradation of the active material [82].

In another paper, Kannan et al. developed humidity gas sensors using a ZnO thin film. The active material was obtained via reactive DC magnetron sputtering, as the deposition was conducted in a gas mixture of Ar and oxygen (1:1). The substrate temperature was set at 300 °C. Through SEM, it could be observed that the surface of the active material presented porous characteristics and spherical particulates with sizes of about 100 nm. To test the gas sensing properties of the sensor, the device was subjected to different humidity levels. As humidity increased, the response increased as well, reaching an astounding response of 42,678, coupled with an excellent response time (3 s) and recovery time (12 s). When exposed to a low humidity of 6.3%, the ZnO thin film presented a resistance of $4.78 \times 10^{11} \Omega$, and when it was exposed to a high humidity of 84%, the sensor's resistance decreased by 4 orders of magnitude and reached a value of $1.11 \times 10^7 \Omega$. The operating temperature of the sensor was not disclosed [83]. P.K. Kannan et al. also employed DC magnetron sputtering to develop ZnO thin films for CO₂ chemoresistive sensors that were thermally treated at 450 °C in an air atmosphere post-deposition. This report examined the effect of film thickness on gas sensing properties. The sample that presented the lowest thickness of 40 nm had the best response, with a maximum sensitivity of 1.13%, and it managed to respond and recover in an equal amount of time of 20 s [84].

Through the pulsed laser deposition method, M. Stamataki et al. synthesized ZnO thin films at two different oxygen pressures of 20 and 40 Pa. All of the other parameters were kept the same. Thus, a study of how oxygen pressure impacts the gas sensing performance was conducted. Moreover, this study focused on how annealing affects the sensor's performance. The samples were annealed for 70 min in air at 300 °C. Through XRD and AFM, it was determined that the thin films underwent structural changes after they were subjected to post-processing heat treatments. The ZnO thin films presented enhanced crystallinity and a smoother surface as a result of the annealing treatments. The sensors were exposed to different concentrations of H₂, and the gas testing measurements revealed that the annealed samples exhibited slower response and recovery times, given the fact that the as-deposited ZnO thin films presented smaller grain sizes, an aspect that improves gas sensing capabilities, as noted in the following results. The as-deposited ZnO thin films exhibited the best response at a temperature of 230 °C with a 99% change in resistance. Increasing the temperature from 150 °C to 230 °C reduced the response time for both thin films, with both films responding to the gas of interest in 24 s. The increase in temperature for the annealed thin films resulted in a slightly lower response to the gas of interest compared to the as-deposited ones [85].

Using RF magnetron sputtering, A.I. Khudiar et al. synthesized ZnO thin films for CO₂ gas sensing, which were then subjected to a post-processing heat treatment at different temperatures (350, 550, 850 °C) in an air atmosphere for 2 h. XRD and AFM analyses revealed that increasing the temperature during the annealing process resulted in the modulation of the active surface area through the modification of grain sizes as a result of the coalescence phenomenon during annealing. Gas testing measurements were conducted in the temperature range of 50 to 350 °C, with the sensor's sensitivity reaching maximum values at 275 °C. The best results were noted for the sample that was annealed at 850 °C with a change in resistance of ~93%, coupled with fast response (20.1 s) and

recovery (21.5) times. In this case, bigger grain sizes resulted in improved gas sensing properties [86].

In a study, C.-Y. Chi et al. prepared AZO (ZnO:Al₂O₃ = 98:2 wt%) thin films and tested the response of the obtained films to formaldehyde (HCHO). The active material was synthesized via reactive RF sputtering, with the flow of oxygen set at 30 sccm. The films were also subjected to post-processing, as they were thermally treated at 400 °C under O₂ pressure for 30 min. The gas testing measurements showed a strong response of 95.5 to a low concentration of 20 ppm of formaldehyde. Moreover, the active material successfully responded to the gas of interest in 60 s, and it recovered to its baseline values of resistance in a short time of 26 s. Those values were obtained at the operating temperature of 350 °C [87]. In another RF magnetron sputtering experiment, M. Zhang, Y. Tang, X. Tian et al. deposited AZO thin films through co-sputtering. Using different RF powers, they obtained thin films with varying concentrations of Al₂O₃ in the active material (ranging from 0 to 22.12 wt%). When exposed to the gas of interest (formaldehyde), the device that presented the best results in terms of sensor sensitivity was the thin film of AZO that contained 11.44 wt% of Al₂O₃. At the operating temperature of 240 °C, the response recorded at 100 ppb of HCHO was 1.43, but it presented long response (423 s) and recovery (409 s) times. The sample that exhibited the best gas kinetics, in terms of successfully adsorbing and desorbing gas molecules in less time, was sample 2 (6.99 wt% Al₂O₃), as it returned to the baseline resistance value in 206 s at 250 ppb formaldehyde [88]. Table 2 compiles information on the deposition method utilized for the synthesis of the ZnO and AZO thin films and gas sensing parameters that define the gas sensing efficacy of the chemoresistive devices.

Table 2. Values of relevant parameters for determining the performance of ZnO and AZO thin films.

Material	Deposition Method	Target Gas	Concentration	Operating Temperature	Response	Response Time (s)	Recovery Time (s)	Ref
ZnO	E-beam evaporation	O ₃	~450 ppb	RT	~5.1 (R)	<2	<15	[82]
ZnO	DC reactive sputtering	H ₂ O (Humidity)	6.3%→84%RH	-	42,678 (S)	3	12	[83]
ZnO	DC reactive sputtering/anneal	CO ₂	1000 ppm	300	1.13% (S%)	20	20	[84]
ZnO	Reactive PLD	H ₂	30,000 ppm	230	~99.5% (S%)	24 (0.4 min)	-	[85]
ZnO	RF magnetron sputtering	CO ₂	-	275	~93% (S%)	20.1	21.5	[86]
AZO (2 wt% Al)	RF sputtering	Formaldehyde (HCHO)	20 ppm	350	95.5 (SR)	60	26	[87]
AZO (11.4 wt% Al)	Magnetron co-sputtering	Formaldehyde (HCHO)	250 ppb	240	1.53 (Ra/Rg)	423	409	[88]
ZnO	Pulsed laser deposition	NH ₃	5.2	300	89	60	65	[89]
AZO	RF magnetron sputtering	CO	-	100	1.048 (Ra/Rg)	<1	-	[90]
AZO (3 wt% Al)	RF magnetron sputtering	CO ₂	-	RT	-	20	-	[91]
ZnO	RF reactive sputtering/annealed	Acetone	500 ppm	400	~100% (S%)	-	-	[92]
ZnO	RF magnetron sputtering	Ethanol vapor	400 ppm	250	~20 (Ra/Rg)	120–240	120–240	[93]
ZnO	PLD/annealing	H ₂	100%	50	>1.6 (Rg/Ra)	-	-	[94]

ZnO	Electron beam evaporation/anneal	H ₂	40 ppm	400	59 (S)	102	-	[95]
ZnO	Pulsed laser deposition	NO ₂ /NH ₃	10 ppm	22	3.3/0.67	200/100	210/250	[96]
ZnO/Ag	Magnetron sputtering/anneal	NH ₃	300 ppm	300	85.74 (S%)	60	-	[97]

3.3. CeO₂

N. Ramshanker et al. investigated the influence of film thickness on the gas sensing properties of cerium dioxide (CeO₂) thin films. The films were synthesized using an in-house RF magnetron sputtering system at a substrate temperature of 400 °C and a power of 75 Watts. By varying the deposition time from 80 to 315 min, the researchers obtained films with thicknesses ranging from 90 nm to 360 nm. The gas sensing performance was evaluated by exposing films of varying thicknesses (90 nm, 150 nm, 220 nm, 260 nm, 320 nm, and 360 nm) to oxygen. Surface topography studies were done using AFM, and an increase in grain sizes was observed with the increase in film thickness, which, in turn, modified the surface roughness as well. Through XRD, it was determined that at a thickness of 260 nm, the grain size increase saturated, and thicker films resulted in smaller grains past this point. Thus, the sample with an optimal thickness of 260 nm exhibited the best gas response, reaching a value of 12.6. This same film also presented the lowest resistivity, at approximately 1 GΩ. The sensor's optimal operating temperature was found to be 400 °C. To test the sensor response, the CeO₂ thin film was first exposed to pure N₂ and then to pulses of oxygen at different concentration values ranging from 100% to 1% (equivalent of 10,000 ppm). The sensor presented a reliable response even to a low concentration of the target gas. Moreover, when exposed to different oxygen concentrations, the material exhibited fast response and recovery times that were constant and reliable. They reported that the CeO₂ sensor presented a response time typically less than 10 s and a recovery time of around 15 s. The sample also underwent selectivity measurements, and it was exposed to 1000 ppm of CO₂, 5 ppm of CO, and 5 ppm of NO_x. The device showed no response to either of those gases, attesting to its highly selective performance towards the gas of interest, O₂ [98]. Using electron beam evaporation, S.M.A. Durrani et al. obtained CeO₂ thin films meant to act as the active material for carbon monoxide sensing. The depositions were conducted at room temperature, and the synthesized thin films were annealed in air for 2 h at 500 °C. The surface morphology of the ceria thin films was studied using AFM, which revealed a roughness of 2.38 nm. The grains presented a columnar-type microstructure with voids between the grains that could enhance the gas sensing properties of the chemosensing device, as they presented trapping centers for gas molecules. The gas testing measurements were conducted at different temperatures, ranging from 300 °C to 500 °C. The best performance of the device towards the gas of interest, carbon monoxide, was observed at an operating temperature of 390 °C, where a high response of ~5000 was recorded. Moreover, the response and recovery times are quite fast, as successful interaction between the gas molecules and the CeO₂ surface led to a high response within 45 s, and only 25 s were needed to return to the initial resistance values [99]. Using RF magnetron sputtering, Xiaojie Zhu et al. developed a gas sensor meant to detect a volatile organic compound, trimethylamine (TEA). The obtained sensors also prove to be tolerant to humidity, making these sensors suitable for environmental tracking of TEA. Through RF magnetron sputtering, a CeO₂/SnO₂ heterostructure was obtained, with CeO₂ acting as the active material. All of the CeO₂ thin films were deposited under the same conditions; the only aspect that varied in obtaining this heterostructure was the thickness of the deposited SnO₂ thin films, which was achieved by varying the deposition time. The deposition times for SnO₂ were 3, 5 and

10 min; thus, the samples were denoted CeO₂/SnO₂-3/5/10. FESEM imaging revealed that the CeO₂ thin film exhibited a highly porous structure, defined by small grain sizes delimited by crevices and pores. The ceria surface presented a much higher surface-to-volume ratio, as the SnO₂ grains were visibly larger. When subjected to exposure to the analyte gas, TEA, the sample that had the thinnest layer of SnO₂, CeO₂/SnO₂-3 presented the best performance overall. When exposed to 20 ppm at an operating temperature of 200 °C, the change in resistance resulted in a response of 23.9, a better response than the values of 22.5 (CeO₂/SnO₂-3) and 16.7 (CeO₂/SnO₂-10) obtained for the other two samples. Dynamic response measurements further revealed that the CeO₂/SnO₂-3 sample exhibited an enhanced morphology that facilitated better interaction between gas molecules and the thin film's surface. When exposed to 20 ppm of TEA, the sensor responded in 4.7 s and recovered in 8 s. Gas testing also revealed that the CeO₂/SnO₂ sensor presented a low detection limit of 0.04 ppm, making it an extremely sensitive and efficient candidate for TEA detection [100]. In another study, Ramshanker et al. deposited a mixed thin film of CeO₂ and HfO₂ using RF magnetron sputtering. Pellets of HfO₂ were placed on the surface of a CeO₂ target. The number of pellets was changed from experiment to experiment to obtain CeO₂/HfO₂ thin films with different atomic weights of HfO₂. Using EDS, the atomic concentrations of the thin films were determined to range from 6 to 23%. When subjected to gas testing, the samples containing 10.6% and 11.6% Hf yielded the best results. At an operating temperature of 400 °C, both samples responded to the gas of interest (O₂) in only 8 s and recovered in 10 s. Those results are extremely good, as the film was exposed to a high concentration of 10,000 oxygen. When it comes to the recorded sensitivity, the two samples had similar values of 14.82 and 15.10. Through XPS, it was determined that these results were obtained thanks to the oxygen vacancies formed at the surface due to the addition of HfO₂ to the structure of the CeO₂ thin films. Those defects facilitate an enhanced interaction between the gas molecules and the surface of the obtained device [101]. The table below provides information on the deposition methods utilized to deposit the active sensing layer and gas sensing parameters (see Table 3).

Table 3. Gas sensing parameters obtained on CeO₂ thin films.

Material	Deposition Method	Substrate Temperature	Target Gas	Concentration	Operating Temperature	Response	Response Time (s)	Recovery Time (s)	Ref
CeO ₂	RF magnetron sputtering	400	O ₂	100,000	400	12.6	10	15	[98]
CeO ₂	Electron beam evaporation	RT	CO	50,000	390	~5000	45	25	[99]
CeO ₂ /SnO ₂	RF magnetron sputtering	-	TEA	20	200	23.9	4.7	8	[100]
CeO ₂ /HfO ₂	RF magnetron sputtering	400	O ₂	10,000	400	15	8	10	[101]
CeO ₂	Pulsed laser deposition	-	TEA	100	450	20	2	186	[102]

3.4. TiO₂

Via RF magnetron sputtering, K. Khojier et al. synthesized TiO₂ thin films with the aim of obtaining sensors for ethanol vapor detection. This study attempted to reveal how different sputtering powers affect the sensing properties of the active material. To isolate this parameter, all the samples presented a thickness of 200 ± 4 nm. The sputtering power varied from 100 W to 300 W. Gas testing measurements revealed that the sample that presented the best results was a TiO₂ thin film deposited at a power of 200 W. Through AFM, it was determined that this sample presented the highest surface roughness, which

inherently meant that the thin film presented a higher surface to volume ratio, an aspect that allows for the creation of more active sites. The measurements were conducted at room temperature, and the sample was exposed to ethanol at concentrations ranging from 10 to 50 ppm. It could be seen that with an increase in the concentration of the gas, the sensor responded much more quickly to the gas of interest. When exposed to 10 ppm of ethanol, the sensor took 63 s to record a 38% change in resistance. Exposing the active material to 50 ppm of ethanol resulted in a 586% change in resistance, and only 9 s were needed to reach such a response. When tackling the recovery time, increasing the gas concentration presented the opposite effect. When 10 ppm of ethanol interacted with the sensor's surface, only 12 s were needed for the material to fully recover to its initial resistance values, whereas the active material needed 60 s to recover after it was exposed to 50 ppm of ethanol [103]. Using DC reactive magnetron sputtering, P. Dhivya et al. developed ammonia sensors in which TiO_2 served as the active material. All of the parameters were kept constant for all three depositions performed, except for the sputtering power. The depositions were held at three different sputtering powers of 100, 150, and 200 W. Through AFM, it was determined that the surface roughness increased when the deposition was held at higher powers. This structural change is also associated with the difference in film thickness. The sample deposited at 150 W exhibited the lowest resistance among the three samples, and it also responded the best when exposed to the gas targeted in this study, NH_3 . Comparing the responses obtained by exposing the 150 W and 200 W deposited TiO_2 thin films, the sample synthesized at a lower power showed a response of ~800 at 100 ppm, compared to the response of ~500 that the other sample presented. At the same concentration, response and recovery measurements were taken to determine which of the sensors performed better overall. When exposed to 100 ppm NH_3 , the sample deposited at a power of 200 W needed 28 s to respond and 122 s to fully return to its baseline resistance. On the other hand, the sample synthesized at 150 W required only 12 s to successfully interact with the gas of interest and 63 s to properly desorb the gas molecules it interacted with and reach initial resistance values. Thus, a highly sensitive NH_3 gas sensor was obtained [104]. Another NH_3 sensor was obtained through DC magnetron sputtering in a study developed by P. Dhivya et al. This paper examined how the substrate bias voltage affects the gas sensing properties of the active material. Thus, TiO_2 thin films were deposited at three different substrate bias voltages: floating potential, -100 and -200 V. The surface of the thin film was relatively uniform across the substrate, as observed in FESEM micrographs. Spherical grains with varying dimensions were observed (20–40, depending on the sputtering power utilized). Using XRD, it was determined that, with different substrate voltages, the thin films changed their crystallinity. At floating potential, there were two peaks corresponding to the anatase and rutile phases, with the anatase peak presenting a higher intensity. With increasing substrate bias voltage, the anatase peak decreased for the film deposited at -100 V, and it even disappeared at -200 V. When subjected to gas testing, the samples were exposed to NH_3 concentrations ranging from 5 ppm to 100 ppm. The thin film deposited with a substrate bias voltage of -100 V exhibited a really high response of 7857 to NH_3 at a concentration of 100 ppm. The sample deposited at -200 V also presented a remarkable response of 1333, but nonetheless smaller than its counterpart. Response and recovery time tests revealed that the -100 V deposited sample also presented better results when it came to the efficient adsorption and desorption of gas molecules. The active material needed 34 s to reach a change in resistance of approximately five orders and 90 s to successfully recover to initial resistance values, making for a highly sensitive and fast-responding NH_3 gas sensor [105]. In another DC magnetron sputtering experiment, TiO_2 thin films were synthesized using different deposition times, ranging from 10 to 40 min in increments of 10 min, thereby varying the films' thicknesses. Field emission scanning

electron microscopy revealed that, with increased thickness, the thin films exhibited better-defined grain boundaries, resulting in greater porosity. This usually facilitates better performance in surface-interaction-driven devices. This was later confirmed when the active material was subjected to NH_3 gas testing. The thin film deposited for 40 min presented the best response and recovery times. When exposed to 100 ppm of NH_3 , the sensor responded in 21 s, reaching a maximum response of 1568, and needed only 58 s to fully desorb the gas molecules from the active material's surface. Selectivity measurements were also performed, and the developed sensor presented a much higher response to ammonia compared to ethanol, methanol, acetone, toluene, and formaldehyde [106]. Through pulsed laser deposition, Nagmani et al. synthesized TiO_2 thin films for H_2S detection. The active material was deposited under the same conditions; the difference between the devices tested was the materials used: strontium titanate, polycrystal Al_2O_3 and silicon (Si/SiO_2). The film deposited on Si/SiO_2 exhibited the best response among all devices obtained, recording a 112,000% change in resistance. Although it exhibited an ultra-high response, the sensor needed 5 min to fully recover to its initial resistance values. The response time to 50 ppm of H_2S was relatively fast, reaching a maximum response in 48 s. The TiO_2 -based sensor also presented a remarkable 85% change in resistance when exposed to only 100 ppb of H_2S [107]. In Table 4, we present a compilation of data regarding the deposition method and the gas sensing results obtained with various chemoresistive sensors utilizing TiO_2 as the active sensing material.

Table 4. Summary of reported TiO_2 thin films obtained through PVD methods for gas sensing.

Deposition Method	Substrate Temperature	Target Gas	Concentration	Operating Temperature	Response	Response Time (s)	Recovery Time (s)	Ref
RF magnetron sputtering	600	$\text{C}_2\text{H}_6\text{O}$	10/50	RT	38/586	63/9	12/60	[103]
DC magnetron sputtering	-	NH_3	100	RT	532	12	63	[104]
DC magnetron sputtering	-	NH_3	100	RT	7857	34	90	[105]
DC magnetron sputtering	-	NH_3	100	-	~1568	21	58	[106]
Pulsed laser deposition	600	H_2S	50	100	112,000	48	300	[107]
DC magnetron sputtering	RT	NH_3	500	250	-	90	110	[108]
DC magnetron sputtering	-	NO_2	66	250	49	~20	~60	[109]
DC magnetron sputtering	-	Alcohol	300	250	1.73	2	~14	[110]
RF magnetron sputtering	-	H_2	2000	450	60	30	30	[111]
DC magnetron sputtering	280	$\text{C}_3\text{H}_6\text{O}$	1000	275	8.22	-	-	[112]

3.5. NiO

In one study, S. Srivastava et al. synthesized a NiO thin film by employing RF magnetron sputtering. The deposition was conducted at a pressure of 3.5×10^{-6} Torr, with the substrate being kept at room temperature. The film did not undergo any other post-deposition processing treatments. FESEM imaging revealed that the morphology of the NiO thin film was highly impacted by that of the underlying alumina substrate. The resulting microstructure was observed to be really porous, with varying crystallite sizes. Gas testing measurements were conducted at different operating temperatures, ranging from 200 to 425 °C. Dynamic sensor response curves showed that when the temperature of the sensor increased, there were considerable improvements in the sensor's ability to quickly respond to and recover from the gas of interest. The sensor presented the highest response to H_2S at a working temperature of 400 °C, with a response value of ~28.8. At the same temperature, the sensor needed 108 s to reach the aforementioned response, and it needed a shorter amount of time to recover, specifically 47 s. Selectivity measurements were conducted to determine the sensors' ability to exhibit greater responses to the gas of interest, H_2S . The active material was exposed to four other gases (NH_3 , NO, CO,

C₂H₅OH). At a lower concentration of 100 ppm of H₂S, the NiO thin film still showed a better response to the gas of interest, with a value of ~10, compared to the responses to the other gases that were tested (all of which presented responses <2.5). Moreover, the limit of detection (LOD) for this sensor was recorded at 135 ppb of H₂S, confirming that a highly selective and sensitive NiO sensor was obtained [113]. To improve the gas sensing properties of the obtained thin film, S. Srivastava et al. conducted a further study on how film thickness and oxygen vacancies affect the gas sensing performance of RF-magnetron-sputtered NiO thin films for H₂S gas sensors. The parameter varied during the deposition process was deposition time, and three thin films were synthesized. The deposition times were 20, 30, and 40 min (with the samples named NO-20, NO-30, and NO-40), yielding film thicknesses of 25 nm, 52 nm, and 76 nm. A multitude of analyses were conducted on the thin films obtained. XRD analysis indicated that NO-30 presented better crystallinity compared to its counterparts. FESEM graphs showed that film thickness did not impact the highly porous and dense microstructure of the thin film. Photoluminescence revealed that the NO-20 and NO-40 thin films presented a lower defect density, which was later confirmed by XPS analysis. When later subjected to gas testing, it was revealed that the higher defect content of the NO-30 sample facilitated a better response than the other two samples. Dynamic curves were obtained under varying temperature and gas concentration conditions. The tests led to the conclusion that the optimal operating temperature for the NiO-based device was 275 °C, where it presented a high response of 260, coupled with an approximate fast response/recovery time of 52 s/23 s [114]. In another article by the same authors, further studies examined how the O₂/Ar gas ratio during deposition impacts the response of the NiO sensor. The deposition method used to synthesize thin films was reactive DC magnetron sputtering. Two thin films were obtained: one in which O₂ represented 10% of the gas introduced in the room during the deposition, and the other sample was deposited in the presence of 20% O₂ (the samples were denoted NO-10 and NO-20, in accordance with the amount of oxygen introduced during deposition). The film deposited at a higher O₂ ratio exhibited higher crystallinity, with a single dominant peak corresponding to the (200) plane. Using field emission scanning electron microscopy, it was determined that the second sample had a larger surface area, as the nanocrystalline grains had small cracks between them, thus creating trap centers for H₂S gas molecules and promoting better gas sensing performance. When subjected to gas testing, the NO-10 sample exhibited a higher operating temperature of 375 °C, with a maximum response of ~8.9 and a response/recovery time of 195 s/56 s. The NiO film denoted NO-20 managed to successfully respond and recover much faster, in 35 s and 14 s, respectively. The maximum response also exceeded that of the NO-10 sample, reaching a sensitivity of ~14.8 at a lower operating temperature of 350 °C [115]. In another DC magnetron sputtering experiment, M.K. Khalaf et al. deposited NiO thin films for H₂ sensing. The aim of this paper was to study how discharge voltage affects a thin film's structure and, therefore, its sensing properties. The discharge voltage was varied from 1.8 to 2.5 kV. Using AFM, it was determined that as the discharge voltage increased, the surface roughness increased slightly, thereby modifying the active surface area. Moreover, the XRD results revealed that the films presented higher crystallinity when the discharge voltage was increased. When exposed to the gas of interest, H₂, the films deposited at 2.5 kV presented the best results among all the depositions. When exposed to a concentration of 3.75% H₂, the sample presented a short response time of 36.5 s and a recovery time of 90.1 at an operating temperature of 300 °C. When exposed to a higher concentration of H₂, the sensor responded more quickly (25 s), but it took much longer to desorb the gas molecules, resulting in a recovery time of 124.3 s. The sensor was also tested at a lower temperature of 275 °C, but it presented similar response times and slower recovery times (25.25 s/140 s and 18.75 s/315.8 s when exposed to 3.75% and 7.5% H₂).

Thus, it was determined that the optimal operating temperature for this sensor is 300 °C [116]. Using the RF magnetron sputtering deposition method, R. Kumar et al. obtained NiO thin films for O₃ detection. The active material was deposited at substrate temperatures ranging from 100 °C to 400 °C, while all other parameters were held constant. Thus, they aimed to study how the substrate temperature during deposition affects the structure of the thin films. All of the deposited thin films presented triangular-shaped grains, as observed in FESEM images. Increasing the deposition temperature led to an increase in grain sizes. Moreover, other flower-like nanostructures were observed scattered across the surface. The best responses were obtained on the sample deposited at a substrate temperature of 200 °C, as it presented the best structural properties. When exposed to a low concentration of ozone (70 ppb), the sensor response recorded at the operating temperature of 200 °C was 12.3, making it extremely sensitive to the gas of interest. Although it presented a great response, the recovery and response times for such low gas concentrations were extremely long, with the sensor needing 3615 s to respond and 2040 to successfully desorb the gas molecules and reach baseline resistance. Further testing was performed on two other compounds, ethanol and acetone, and the results showed that even at higher gas concentrations, the response was lower than the initial response to O₃. The response to 100 ppm of ethanol was 1.8, and the response of the NiO thin film to 25 ppm of acetone was 0.8. Even though they showed lower response values, they managed to respond and recover much faster, but still quite slowly (675 s and 300 s to ethanol and 180 s and 170 s to acetone) [117]. Table 5 compiles information on the deposition methods used to synthesize the gas sensing layer and details about the operating temperature, concentration, response, and response/recovery times of NiO-based chemoresistive gas sensors.

Table 5. Reported results of NiO thin films deposited through PVD methods used in gas sensing applications.

Deposition Method	Substrate Temperature	Target Gas	Concentration	Operating Temperature	Response	Response Time (s)	Recovery Time (s)	Ref
RF magnetron sputtering	RT	H ₂ S	200	400	28.8	108	47	[113]
RF magnetron sputtering		H ₂ S	50	275	260	52	23	[114]
DC magnetron sputtering	RT	H ₂ S		350	14.8	35	14	[115]
DC magnetron sputtering	-	H ₂	-	300	~1750	36.5	90.1	[116]
RF magnetron sputtering	200	O ₃	70 (ppb)	200	12.3	3615	2040	[117]
RF magnetron sputtering	200	Ethanol	100	400	1.8	675	300	[117]
RF magnetron sputtering	200	Acetone	25	400	0.8	180	270	[117]
DC magnetron sputtering	RT	Ethanol	5	250	~6	167	99	[118]
DC magnetron sputtering	250	NO ₂	50	200	46	-	-	[119]
Pulsed laser deposition	200	H ₂	30,000	125	76	900	600	[120]

3.6. SnO₂

When researching metal oxide chemoresistive gas sensors, SnO₂ is the material that is most commonly employed for such applications [121]. According to a survey conducted on Web of Science dating back to 2022, the most searched articles in this area of study were on chemosensing devices based on tin oxide [122]. This is due to the fact that this metal oxide presents improved properties compared to other materials in the same category. For example, SnO₂ has the capability of adsorbing oxygen species more strongly, as it can create more oxygen vacancies thanks to its dual valence states, making it a strong contender in gas sensing applications [123,124]. As with most metal oxide-based chemoresistive sensors, its other advantages include high sensitivity, conductivity, chemical stability, and, more importantly, its cost-effectiveness. Regarding the issues experienced with SnO₂ gas sensors, they present poor selectivity and require high

operating temperatures, leading to problems with power consumption [125–127]. The current focus is on finding affordable ways to solve these problems. This section focuses on pristine SnO₂ gas sensors produced by PVD methods.

The gases primarily targeted for detection using SnO₂-chemoresistive-based sensors are hydrogen (H₂) and nitrogen dioxide (NO₂). In a recent article from 2022, Yijun Yang et al. employed RF sputtering to deposit tin oxide on the surface of a glass wafer, and Au electrodes were deposited using E-beam evaporation, creating a two-terminal device. This study examined how annealing affects the structure of thin films and how tuning the temperature and duration of this process can lead to improved gas kinetics. The obtained SnO₂ thin films were thermally treated for up to 5 h at temperatures ranging from 300 to 900 °C, with gas sensing measurements revealing that the devices annealed at 500 °C for 3 h exhibited the best gas sensing performance. At this temperature, the grain morphology of SnO₂ changed, leading to a change in surface roughness, as confirmed by AFM analysis. Moreover, using XRD, the grain sizes were calculated, and it was revealed that they increased in size in accordance with the time they spent under treatment. This resulted in a more porous surface of the tin oxide thin film, which, in turn, translated to improved gas dissociation kinetics. This theory was confirmed in this paper by the fact that the as-deposited thin film presented a 251% change in resistance, while the annealed sample recorded a considerable increase in response of up to 378%. When exposed to a concentration of 50,000 ppm of H₂ at an operating temperature of 300 °C, the sensors' response and recovery times were around 12 and 53 s, respectively. Selectivity measurements were also conducted towards CO₂, NH₃, and NO₂, and the results indicated an improved response towards the gas of interest [128].

In a recent study with a similar approach, Vipin Kumar et al. synthesized SnO₂ thin films for hydrogen detection. Compared with the previous study, in their research, the thin films were annealed for 6 h at a temperature of 450 °C. The thin film exhibited a highly uniform, porous surface with a nanogranular morphology. Moreover, visible cracks were dispersed on the surface of the active material. Although different annealing conditions were used in this study, the device's operating temperature remained at 300 °C. At a low concentration of 50 ppm, the device presented a maximum response of 3.04. When comparing this device to the previously discussed one, the recovery time is similar, at only 52 s. Still, the time needed to respond to hydrogen is slightly longer, at 42 s. Although the annealing parameters differ significantly, the results are quite similar. This is explained by the differences in the deposition parameters used. This study also reports a very low limit of detection (LOD), with the sensors producing a reliable response for a concentration of only 175 ppb [129]. In another study targeting hydrogen, M. Abinaya successfully synthesized SnO₂ thin films that enabled the development of a highly sensitive device capable of producing a reasonable response at room temperature, given that tin oxide films often require high operating temperatures. Three samples were synthesized by varying the cathode power, thereby highlighting how sputtering power impacts the structure of thin films. A large active surface area was observed in the provided FESEM images, with small grain sizes of ~50 nm. Moreover, considerable grain boundaries were observed, thus affecting the electrical conductivity of the tin oxide film. Through X-ray diffraction, it was determined that increasing the sputtering power resulted in an increase in crystallite size and stoichiometry, leading to more porous films. Moreover, fewer defects, lower strain and less stress were observed as the cathode power increased. The SnO₂ sensors were exposed to H₂ at concentrations ranging from 20 ppm to 100 ppm. At room temperature, the sample deposited at 70 W showed the best response among the three samples obtained, with a value of approximately 4.7 when tested towards 100 ppm H₂. At the same concentration of analyte gas, the sensor required 34 s to reach a 90% change in resistance and 84 s to successfully return to its baseline. Stability analysis

was performed over 30 days, with the device tested every 5 days, and it showed no change in baseline resistance and gas sensing properties [130].

Another analyte targeted in the development of SnO₂-based chemoresistive devices was NO₂. In their research, Margus Kodu et al. successfully developed tin oxide thin films through the pulsed laser deposition method and obtained SnO₂ sensors that function at room temperature. During the deposition, a KrF excimer laser with a wavelength of 248 nm was employed, with the substrate kept at room temperature. An SEM image revealed a highly porous surface, with grain sizes of approximately 15 nm. Moreover, the grains formed networks of thin cracks between them, presenting possibilities to trap gas molecules and improve response. Through XRD and Raman spectroscopy, it was determined that the obtained thin films presented an amorphous structure. The sensor baseline was measured in synthetic air at 21% relative humidity, and the NO₂ concentration introduced into the reaction room was 4 ppm. The aim of this research was to study how the bias affects the sensor's response. It was observed that changing the bias voltage two times increased the response of the sensor by a factor of 25. The operating bias for the SnO₂ sensing device that they obtained was 4 V, at which it recorded an astounding response of 7730, coupled with a response time of 3 s. The recovery time ranged from 27 to 43 s, depending on the concentration of NO₂ to which the device was exposed. This study also touched on how the electrode gap affects the gas sensitivity of the device, specifically that decreasing the gap leads to an improvement in sensor response [131] (see Figure 5).

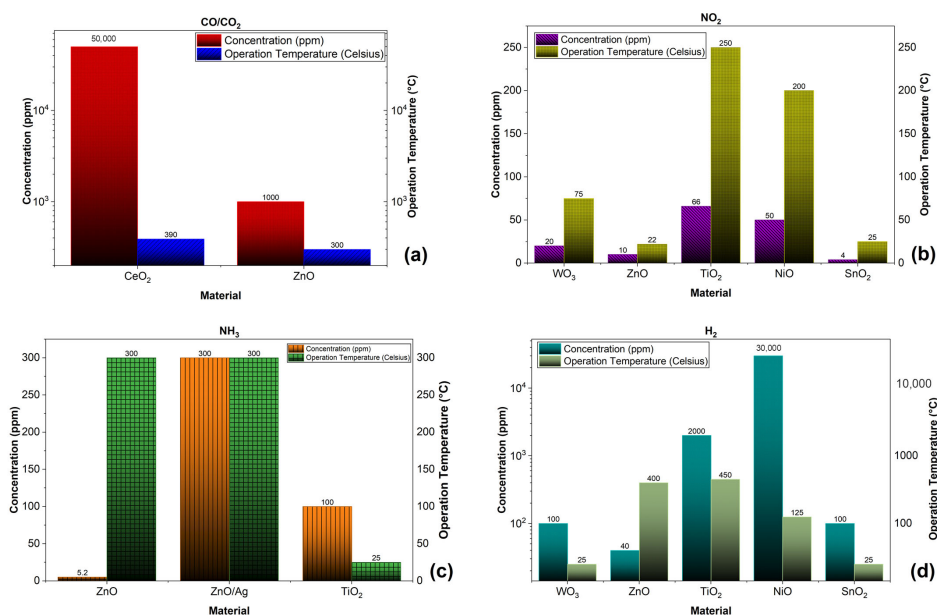


Figure 5. Plotted graphs with respect to the results found within the articles. This figure contains the operation temperature and gas concentration of the materials required to detect (a) CO/CO₂ [84,99], (b) NO₂ [76,96,109,119,131], (c) NH₃ [89,97,104], and (d) H₂ [77,95,111,120,130].

Using the RF sputtering deposition technique, Anjali Sharma et al. obtained SnO₂ thin films, which they employed for NO₂ detection. The main focus of their research was to explore the influence of the growth pressure used during the deposition, which they varied from 12 to 18 Torr. Post-deposition, all of the samples were annealed for 3 h at 300 °C. The initial SnO₂ thin film was amorphous, but after the thermal treatment, it presented improved crystallinity. When comparing the diffractograms, the peaks showed a decrease in intensity as the sputtering pressure increased. Moreover, the crystallite size decreased

with increasing deposition pressure, ranging from 8 to 13 nm. This pressure-dependent characteristic was further confirmed by the grain size determined through AFM. This aspect translates into an active material with a large surface area, which creates more sites for NO₂ gas molecules to interact, leading to an increased response even at lower temperatures. Their findings propose a low-temperature operating sensor, with incredible responses and moderate response and recovery times at 100 °C [132]. Table 6 compiles all the information about the deposition processes utilized in the synthesis of the active sensing material of SnO₂-based chemosensors presented in this subsection, coupled with their respective gas sensing results.

Figures 5 and 6 effectively compile data from the results reported in this review article in order to present a comparative visualization of various results obtained with the different metal oxides employed as active sensing layers in chemoresistive gas sensor applications.

Table 6. Summary of reported SnO₂ thin films obtained through PVD methods for gas sensing.

Deposition Method	Substrate Temperature	Target Gas	Concentration	Operating Temperature	Response	Response Time (s)	Recovery Time (s)	Ref
RF magnetron sputtering	-	H ₂	50,000	300	257.34%	12	53	[128]
RF magnetron sputtering	RT	H ₂	50	300	3.04	42	52	[129]
DC reactive magnetron sputtering	-	H ₂	100	RT	4.7	34	84	[130]
Pulsed laser deposition	RT	NO ₂	4	RT	7730	3	43	[131]
RF diode sputtering	200	NO ₂	50	100	30,000	96	2562	[132]

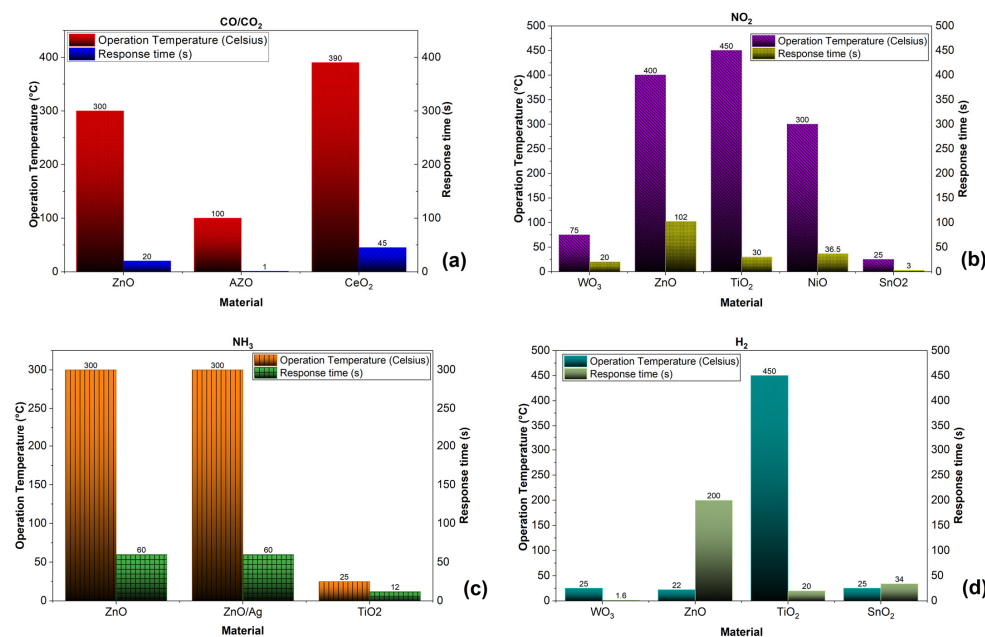


Figure 6. Plotted operation temperatures and response times for different materials targeted in this study for the detection of (a) CO/CO₂ [84,90,99], (b) NO₂ [74,95,111,114,131], (c) NH₃ [89,97,104], and (d) H₂ [77,96,111,130].

4. Strategies for Enhanced Detection Through Functional Property Tailoring of Oxide Thin Films

4.1. Doping and Strain Engineering

In a world where tracking the compounds in our environment becomes increasingly important, it is of utmost importance that the devices developed for these applications

present the best performance. As it is generally known, when working with chemoresistive gas sensors, more often than not, metal oxide-based devices will present a couple of issues worth mentioning, most importantly, poor selectivity and high operating temperatures [133]. To tackle this problem, researchers have come up with innovative ways to improve these types of sensors. One such way is through doping the active material with different elements. Doping can achieve improvements in these characteristics by either electronic or chemical sensitization and, moreover, by structural changes induced by dopants within the material or on its surface [134–141]. Through electronic sensitization, we understand that introducing dopants can induce a shift in the local Fermi level of the metal oxide, influencing its resistance [136,139,141]. For example, doping aliovalent cations with a different valency than the host material allows us to control the carrier density [141]. Another form of electronic sensitization can be given by the formation of Schottky barriers between noble metal dopants at the interface with the metal oxide, resulting in thicker electron-depletion layers or hole accumulation layers, depending on the nature of the material (if it is either n-type or p-type) [137]. In terms of chemical sensitization, doping the active material with transition metals and noble metals improves the reaction towards different gases, offering a solution to selectivity problems [138]. Those elements act as catalysts; hence, they have increased reactions with different analyte gases. Lastly, doping can lead to physical changes within the material, specifically the creation of more oxygen vacancies, which, in turn, provide additional charge carriers for redox reactions [139,140].

Focusing the framework back to PVD-synthesized thin films, we herein report erbium-doped SnO₂ thin films deposited by electron beam deposition. Firstly, Manreet Kaur Sohal et al. synthesized SnO₂ nanoparticles using a low-cost co-precipitation method in order to accurately vary the atomic weight of Er. After their synthesis, the nanostructures underwent post-processing, during which they were dried in an oven, ground, and then calcined in air for two hours. Multiple samples underwent testing: pristine SnO₂ films and three Er-doped SnO₂ thin films with Er dopant ranging from 1 to 5 wt%. Their thicknesses were also varied between 50 and 150 nm. Imaging of the surfaces using FESEM showed that increasing the doping element improved morphology for gas sensing applications. While the undoped films presented a homogeneous structure with considerable voids separating the grains, the grains on the sample with the highest dopant were agglomerated, and their surface had smaller particles. Through XRD, it was determined that the presence of Er in the structure of the thin film resulted in crystallinity changes, with the crystallite sizes increasing with the atomic weight of the dopant. This confirms that Sn⁺⁴ ions are replaced by Er⁺³ ions due to their greater ionic radii. Through Raman spectroscopy, it was determined that the addition of Er into the lattice structure of the thin films resulted in an increased number of oxygen vacancies within their structures. This further enhanced the interaction with oxygen species, leading to an increase in the electron depletion layer, which, in turn, translated into better gas sensing properties. The sample that presented the best gas kinetics had a thickness of 100 nm, and the atomic weight of Er was 3%. When exposed to various gases at a concentration of 1 ppm, this sample presented a really selective behavior towards NO₂ and also moderate response and recovery times at room temperature (18 and 24 s). The sensor also managed to reliably respond to low concentrations of only 15 ppb of NO₂, attesting to a highly sensitive room-temperature chemosensing device [141].

In a recent study, Marwen Mezyen et al. developed SnO₂ thin films doped with dysprosium, a rare earth element, through RF magnetron sputtering. The active material was deposited on glass and alumina substrates that featured Pt interdigitated electrodes that were screen-printed. The atomic weight of Dy was varied from 1 to 7 wt% by mixing SnO₂ and Dy₂O₃ powders to obtain the targets used during the magnetron sputtering

experiment. The deposition itself lasted 3 h, and an RF power of 200 W was used. Using FESEM, the surface morphology was studied, revealing differences between the films deposited on the two different substrates. The nucleation process on the alumina substrate led to the synthesis of a thin film containing numerous microaggregates with small grains between them, thereby creating a dense surface with a high active surface area. AFM revealed that Dy doping impacted grain size: increasing the doping element resulted in a decrease in grain size. Through XRD, it was determined that increasing the dopant decreased the peak attributed to SnO_2 , therefore attesting to the successful integration of dysprosium into the thin film. Similarly, with Er, the ionic radius of Dy^{+3} ions is greater than that of Sn^{+2} , causing Dy to occupy substitutional positions and inherently modify the lattice structure of SnO_2 . When assessing the gas sensing performance of the synthesized thin films, the structure with an atomic weight of 7% exhibited the best sensing properties. The improvement in response was attributed to Dy acting as a defect promoter, thereby increasing the density of oxygen vacancies. As with Er, this defect engineering of the active material modulated the band structure and, in turn, resulted in a thicker electron depletion layer. The response to NO_2 showed over a 42% change in resistance at an operating temperature of 250 °C, with the response improving to 63.6% after one year of testing. This improvement in performance is associated with aging, in which oxidation over time alters the distribution of oxygen vacancies distributed on the surface of the active material, thus increasing the active sites available for gas molecules. Although it presents a lower operating temperature than most pristine SnO_2 -based chemoresistive gas sensors, the response and recovery times are on the moderate side, as the sensor needed 9 min to fully respond to the analyte gas and 5.55 min to successfully desorb gas molecules [142]. Figure 7 shows the dynamic response curves of ZnO-based chemoresistive devices doped with Ga, Al, and Ca towards acetone, ethanol, methanol, and formaldehyde.

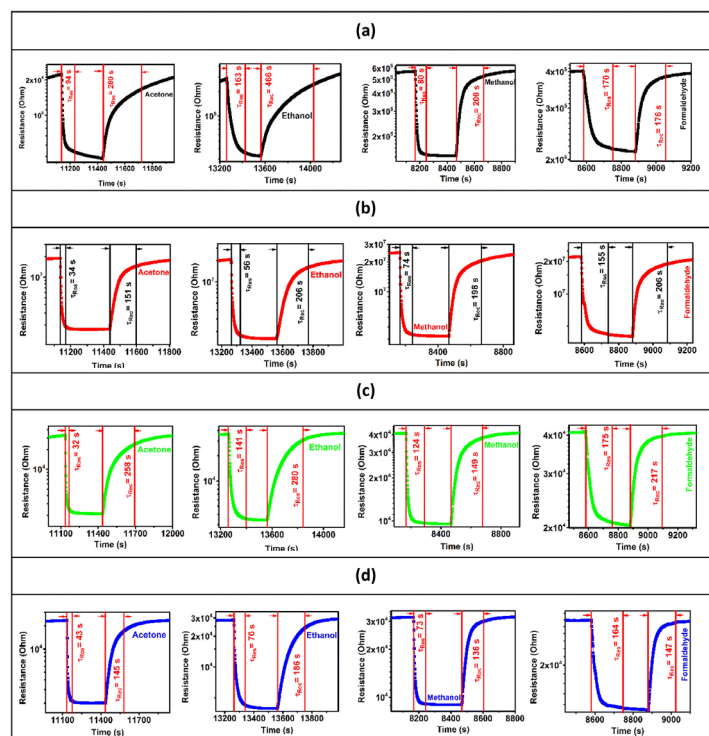


Figure 7. Response and recovery times of (a) pristine ZnO-based, (b) Ca-doped ZnO-based, (c) Al-doped ZnO-based, and (d) Ga-doped ZnO-based chemoresistive sensors, reprinted with permission from the Royal Society of Chemistry [143].

Another technique often employed to tailor the properties of oxide thin films used for gas sensing is the engineering of strain within the crystal. Strain is usually implemented during material processing through small differences between the lattice parameters of substrates and the deposited thin film or by building heterostructures, meaning the deposition of two or more materials with different lattice parameters of the crystallographic structure. This lattice strain acts as an impetus for the introduction of surface vacancies or structural defects, specifically dislocations, in a controlled fashion, which will alter the mechanism by which gases are detected, improving the material's sensitivity and adding a degree of selectivity. For instance, de Lima et al. studied ZnO films obtained through radio-frequency magnetron sputtering in an oxygen-rich atmosphere from both experimental and theoretical points of view. Their findings showed that when the deposition was performed in an oxygen-rich atmosphere, the obtained films exhibited a distorted structure with Zn-O bond stretching and a selectivity that could be tuned towards ozone detection by controlling the density of defects. The RF power employed during the experiment was 50 W. Silicon and glass substrates were used. The devices underwent thermal annealing in air for 12 h. Through SEM, cross-sectional views of the ZnO films showcased that varying the oxygen concentration inside the chamber resulted in thickness variations in the active material. Moreover, the thinner structures exhibited smaller grain sizes, thereby increasing the active surface area [144]. P. Amézaga-Madrid et al. [145] used an AACVD technique to synthesize ZnO films, and the crystallographic characterization showed the nanocrystalline character of the film, with a high density of grain boundaries and the presence of lattice strain. The films exhibited high activity toward CO₂ exposure when detected at 100 °C, with further improvements in sensitivity and dynamic performance under UV-A irradiation. In another report, Sáenz-Hernández et al. studied the microstructural evolution of tungsten trioxide WO₃ thin films obtained via reactive magnetron sputtering and the impact on the detection of CO₂. The power applied during the deposition was 60 W, while the gas mixture consisted of a ratio of 75% Ar and 25% O₂. Their study was performed on films with different thicknesses, and the results showed that there could also be a negative impact on performance if a larger amount of built-in stress is present, which was corroborated by the surface porosity, grain size, and density of active sites that were observed in SEM graphs and XRD analysis. The thinnest film, at 42 nm with the lowest calculated average maximum strain, showed the highest sensitivity (92%) towards CO₂ detection [146]. Furthermore, in a theoretical report, Singha et al. conducted a comprehensive first-principles investigation of an In₂O₃ monolayer and the effects of different types of strain on the detection of various types of gas molecules. Their findings showed that strain engineering, mainly the introduction of moderate tensile and compressive strains, can be an impactful tool to augment sensing performance and selectivity [147].

4.2. Morphologic and Surface Processing

A different approach to enhancing the gas sensing properties of chemoresistive gas sensors involves modifying the surface of an active sensing material. One pathway to increasing the gas sensing capabilities of metal oxide-based gas sensors involves functionalizing their surfaces using various nanoparticles [133]. Similar to the doping approach, surface functionalization leads to electronic and chemical sensitization, and morphological changes on the surface increase the density of activation sites for molecular interactions [127,148–150]. An example of electron sensitization is the Schottky barrier formed between a noble metal like Pt and a metal oxide. The noble metal nanoparticle has a higher work function than MO, thus creating a barrier that thickens the electron depletion layer [150]. In terms of chemical sensitization, functionalizing a material's surface with nanoparticles that act as catalysts lowers the activation energy, which results

in lower operating temperatures [123,126]. Another phenomenon that occurs after the addition of noble metal to a material's surface is the so-called "spillover" effect, in which molecular oxygen species are dissociated into reactive oxygen species, which leads to a larger density of oxygen ionic species on the material's surface [127,150]. Lastly, the addition of nanoparticles and nanostructures to the sensing materials' surface may modify the surface-to-volume ratio. Higher ratios lead to a higher density of active sites for the molecules to interact with [133].

Focusing our attention on metal oxides synthesized through PVD methods, a recent article delves into how the gas sensing properties of a SnO₂ thin film obtained through sputtering deposition are improved with the addition of RuO₂ nanosheets on the sensor's surface. The thin films had a thickness of 50 nm, and the sputtering experiment was done using a shadow mask. The sensors were fabricated in batches, using multiple deposition methods for the different elements of the device. Electron beam evaporation, plasma etching, photolithography, and silicon wet etching were used to synthesize the membrane, sensing electrodes, and microheater. Scanning Transmission Electron Microscopy revealed the 2D sheet structure of the RuO₂ material, while atomic force microscopy confirmed the sub-nanometric thickness of these nanostructures. The aim of this study was to determine if the addition of RuO₂ nanosheets to the device's active surface improves the response to ethanol. The gas sensing performance of pristine and functionalized SnO₂ was tested at temperatures ranging from 125 to 345 °C. In the case of the modified sample, the optimal operating temperature was found to be 290 °C, where it presented a response of ~10, around 5 times higher than the response recorded for the pristine tin oxide. The improved sensing characteristics are a result of the increased surface-to-volume ratio due to the addition of RuO₂ nanosheets, as more active reaction sites are created. The catalytic effect of these nanostructures is further enhanced by spillover, as partially oxidized ethanol molecules migrate towards the SnO₂ active material after interacting with the nanosheets. Moreover, the RuO₂ nanostructures improve the device's resistance to humidity by dissociating water molecules or forming a hydrophobic layer. To assess sensor stability, the device was tested over 28 days, with 7-day intervals between measurements. Over this period, the SnO₂-based chemosensing device exhibited a stable response of 9.59 towards 10 ppm of ethanol, with values falling within a ±3.7% range. Most importantly, the authors reported that this device enables real-time breath control, thanks to an extremely low limit of detection of ~5 ppb [123]. In another recent study, Amit Kumar Gangwar et al. synthesized Pd/SnO₂ thin films through reactive magnetron sputtering. The deposition of SnO₂ was performed using a gas mixture of 90% Ar and 10% O₂ at 10 mTorr, with alumina as the substrate. The surface of the tin oxide thin film was functionalized with a 5 nm layer of Pd, which served as the catalyst. The deposition parameters slightly varied, as the working pressure was set at 5 mTorr and the DC power was kept constant at 30 W. FESEM imaging revealed a rough surface with cracks. The functionalized sample presented a higher surface area because the Pd grains were smaller than the underlying SnO₂ structures. Besides the chemical and electrical sensitizations that usually impact the enhancement in response when it comes to the functionalization of the active material surface, through Time-Resolved Photoluminescence, it was determined that there was an increase in the oxygen vacancies present compared to the pristine sample. Moreover, XPS analysis confirmed the presence of PdO. This explains the increase in the device's baseline and the improved reactivity towards the gas of interest, carbon monoxide. Regarding gas sensing performance, the Pd-functionalized device outdid the pristine SnO₂ thin film in terms of response and response/recovery times at an operating temperature of 200 °C. The device also offered the possibility of operating at 100 °C, which translates into reduced power consumption [151].

5. Conclusions and Perspectives

Through this review, we aimed to examine the recent advancements in metal oxide-based chemoresistive gas sensors. This study revealed multiple ways in which PVD-produced thin films can be tailored to enhance sensing properties. A consistent finding across the materials targeted in this paper was the impact of the substrate temperature during deposition. It was shown that increasing the substrate temperature caused structural changes in grain size, thereby affecting the specific surface area of the active material. These changes in the structure of the films' surfaces can be controlled to enhance sensors' gas sensing capabilities. Furthermore, our analysis underlined the critical role of defect engineering. This review found that oxygen vacancies present in the thin film structure were an important factor affecting sensor efficacy. It was demonstrated that inducing a higher number of oxygen vacancies in an active material's structure is an effective tailoring approach that improves gas sensing properties. By inducing more vacancies in a film's structure, the baseline resistance of sensors can be modulated so that the gas sensing mechanism is enhanced, thus leading to improved responses. This has been done specifically during magnetron sputtering and PLD experiments, as they allow for changes in the gas mixture and partial pressure. On the other hand, electron beam deposition is less versatile because it requires a high-vacuum environment during synthesis. Besides modifying deposition parameters during the experiments, researchers have employed post-processing methods on the obtained nanostructures through thermal processing. This represents another effective way to tailor the defect density and surface morphology of metal oxide-based chemoresistive sensors.

Delving deeper into our findings, tungsten oxide (WO_3) emerges as an excellent candidate for detecting NO and NO_2 gases. Although the material was synthesized using different deposition methods, WO_3 consistently presented high sensitivity towards nitrogen oxides. Moreover, the obtained devices exhibited very fast response and recovery times, further attesting to the excellent performance of this material towards NO_x . Beyond nitrogen oxides, WO_3 -based chemoresistive sensors also presented remarkable results for CH_4 and H_2 detection. ZnO thin films showed significant promise for the development of CO_2 -targeted gas sensors, delivering remarkable gas sensing performance. Aluminum-doped zinc oxide thin films have often been employed for the detection of VOCs. Two similar studies focused on the detection of formaldehyde by depositing AZO thin films through magnetron sputtering. The sample with a lower aluminum atomic weight exhibited superior sensitivity to the analyte gas and showed better performance overall in terms of response and recovery times. Our findings on cerium oxide (CeO_2) thin films indicate that devices based on this material generally require higher operating temperatures than those of the other materials discussed in this review, as they typically achieve peak performance around 400 °C. Comparing two studies revealed that incorporating HfO_2 into the structure of CeO_2 thin films improved the sensor's response and recovery times to oxygen. Another article included in our review described a device that consisted of a heterostructure of $\text{CeO}_2/\text{SnO}_2$ that targeted TEA, which achieved impressive performance at a low operating temperature of 200 °C, suggesting the possibility of developing CeO_2 gas sensors with lower power consumption. Multiple studies on TiO_2 thin films deposited through DC magnetron sputtering were conducted, and it was revealed that highly sensitive NH_3 gas sensors can be obtained using this structure as the active material. The most notable property of these sensors is their ability to present high and reliable responses to ammonia and also their fast recovery and response times. This represents a great opportunity for future studies and advancements in obtaining cost- and power-efficient sensors. Another remarkable result obtained with this material was reported for ethanol detection at room temperature and for alcohol at an operating temperature of 250 °C, paving the way for the development of

gas sensors for VOCs. Lastly, excellent results were obtained for H₂S detection when employing NiO as the active material in the development of chemoresistive sensors. Its consistent high sensitivity and fast response/recovery times across different operating temperatures make this material a good candidate for future developments in chemosensors for H₂S testing. The interaction between NiO and VOCs was also tested, but it exhibited poor gas sensing performance due to high response and recovery times and low sensitivity. A general remark across all the materials reviewed is that magnetron sputtering is the preferred PVD method for depositing thin films used in chemoresistive gas sensors, owing to its high reproducibility.

A consistent trend observed across the metal oxide sensors in this review is that their primary limitations stem from high operating temperatures and poor selectivity. To address these challenges, we have highlighted recent breakthroughs in tailoring thin film properties to enhance overall sensing performance. The primary strategies for achieving these improvements include:

- Doping;
- Strain engineering;
- Surface functionalization.

Specifically, integrating noble metals via doping or surface modification allows them to function as catalysts. This accelerates the reaction with analyte gases, thereby boosting sensitivity and reducing response times. Furthermore, both doping and strain engineering can induce structural modifications within the metal oxide lattice, most notably the creation of oxygen vacancies. This shift alters the density of charge carriers, which results in a more resistive thin film at baseline, but it significantly amplifies a sensor's response to the target gas. Moreover, the functionalization of metal oxides can lead to a significant change in the limit of detection, thus paving the way for breath control applications. The addition of nanostructures for surface functionalization effectively creates heterojunctions, inherently modifying the charge transport at the surface. Moreover, they act as a catalytic element by dissociating the gas molecules that interact with them, thus leading to improved sensor response. Some results also showed that such functionalization led to improved structural properties when exposed to different humidities, thus paving the way for future developments in more complex practical applications. This statement is also supported by the fact that when functionalizing chemosensing devices through these methods, lower operating temperatures were observed. This represents an effective way to reduce production costs and power consumption of future chemoresistive tech.

Lately, in an attempt to improve the capabilities of current chemoresistive gas-type sensors and gas sensing devices as a whole, a new research direction is also being intensively followed. This is reflected in the development of devices based on sensor microarrays. Inherently, they integrate multiple microsensors that either use entirely different gas-sensitive materials (multimaterial arrays) or different segments of the same material that has either been functionalized or operates locally under different conditions (single-material arrays). Each microsensor interacts with the gas it is exposed to, causing multiple distinct interactions to take place simultaneously. The combined outputs of the sensors form a “vector signal”, and different algorithms or machine learning models are used to process the interactions between the device and different gases [152–154]. This approach has shown promise in developing devices that present high selectivity towards different analytes. Although they have potential, there are serious challenges with developing such devices at the industrial level in the manufacturing process, leading to signal deviation due to sensor displacement. This results in hardware inconsistencies; thus, future studies are needed to successfully reach batch production levels.

While magnetron sputtering remains the standard for industrial-scale manufacturing, pulsed laser deposition (PLD) has recently garnered significant attention. This interest is driven by the development of large-area PLD systems, which enable production upscaling through batch processing. These systems can coat 150 mm wafers with a thickness variation of less than 5%, enabling the fabrication of numerous devices on a single wafer and proving PLD's viability on an industrial scale [155]. For a long time, this synthesis method was considered usable only in laboratory settings, but nowadays, large-area PLD systems address a key limitation of traditional systems by incorporating a “droplet trap” [156]. This component eliminates the particulate formation common in PLD processes, resulting in highly uniform thin films. Since droplets may negatively impact the sensing capabilities of metal oxide films, this innovation is important for gas sensing applications. Finally, these systems feature a target carousel that allows for the deposition of heterostructures. Combined with PLD's ability for stoichiometric transfer, this technology is promising for developing complex oxide-based gas sensors with improved response and selectivity. Preiß et al. used a large-area PLD system to deposit thin films of tin oxide (SnO_2), which demonstrated good performance at operating temperatures between 200 °C and 375 °C. The obtained sensors were tested for Carbon Monoxide (CO) and Nitrogen Dioxide (NO_2), and the lowest concentration to which the device presented a reliable response was 0.5 ppm of NO_2 . Thus, a highly sensitive NO_2 sensor was obtained. The SnO_2 chemoresistive gas sensor also presented relatively fast response and recovery times of approximately 2 min. Moreover, in this article, the PLD method was used to integrate catalytic elements, such as Palladium (Pd). The addition of this element leads to an improvement in baseline stability and also reduces cross-sensitivity towards humidity, thus improving the reliable operation of the device in different environmental conditions [157]. In another article, Preiß et al. report the development of chemosensors based on different materials (CuO , WO_3 , SnO_2 and ZnO). Gas sensing parameters are reported only for devices based on SnO_2 thin films, as the other materials tested did not present reliable responses. Using the tin oxide device, excellent responses were obtained at 300 °C. When exposed to the gas of interest (CO), the device took about a minute to respond reliably [155]. Thus, we can affirm that PLD large-area systems have enormous potential in the future industrial development of high-performant and reliable chemoresistive gas sensors.

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