Electrospun Zinc Oxide Nanofiber Based Resistive Gas/Vapor Sensors - A Review

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Abstract

Resistive zinc oxide (ZnO) sensors play a pivotal role in detecting various gases and vapors due to their high response, low cost, stability, tunability, and simple fabrication. Hence, it is necessary to know the recent status of research in resistive ZnO sensors. The sensitivity is determined by the reactions at the surface of the nanofiber (NF); therefore, the surface area defines the foremost sensor characteristics. Electrospun metal oxide NFs exhibit a high surface area and unique electrical properties that can be tuned, and they are highly sought after as materials for resistive gas sensors. So far, various strategies are adopted to improve the sensitivity and the selectivity of ZnO NFs. This review summarizes the recent methods utilized by various researchers to improve the sensitivity of the ZnO electrospun metal oxide NF-based resistive gas sensors. Also, it discusses the influence of process parameters on the structure and morphology of ZnO NFs, and the mechanism of gas sensing and highlights its improvement through advanced methods. The sensitivity of the NF has been improved through tuning the structure and morphology of NFs and doping. Further, modification of NF sensitivity through functionalization, the addition of carbon nanomaterials, and high-energy irradiation are also discussed. Based on the recent literature, the performance of doped ZnO NF for various gas sensing is highlighted. The outcome of this review gives insight to academic researchers and industry for further investigation and development in resistive gas sensors and their applications.

Keywords: ZnO-NFs; Metal oxides, Electrospinning; Gas sensing mechanism; Gas sensor.

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1. Introduction

The demand for portable, cheap, and effective gas sensors for day-to-day life is increasing. It includes air quality assessment, breath analysis for disease diagnosis, gas leakage detection in the working environment, and hazard monitoring. Even though extensive literature data is available on the metal oxide resistive gas sensors, several obstacles such as cross-sensitivity and low selectivity are yet to be rectified. Additionally, the accuracy of gas sensors depends on operating temperature and humidity.^[1] The response of the resistive gas sensors is commonly measured in terms of change in sensor resistance, and the resistance of metal oxides fluctuates with

changes in temperature. As a result, the accuracy of the sensors can be severely harmed.^[2] Hence, identifying the best-suited material for developing the effective resistive type of gas sensors remains a challenge, and this area is considered an important field of research.

Metal oxide semiconductors are the preferred material for resistive sensors to detect various types of gas. These gas sensors are popular due to their low cost, long-period stability, good response and recovery time, and high sensitivity. Many researchers have carried out their work on different materials such as ZnO,^[3–5] SnO₂,^[6] Co₃O₄,^[6,7] and V₂O₅.^[8–10] To improve the gas molecule interaction in semiconductor surfaces, increase the surface-to-volume ratio, and change the energy band structure and morphology, doping the surface with different materials are common practice in the design of gas sensor material.

Electrospun metal oxide NFs are often used for the fabrication of gas sensors. The high suitability of metal oxide NFs for resistive sensors is due to the high porosity, large surface area, high sensitivity, stability, and low response and recovery time.^[11] Additionally, the flexible nature of the as-

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spun fibers allows molding the fibers to the required final shape before calcination. Additionally, the properties of the NFs can be easily tuned by introducing dopants, incorporating vacancies, forming a composite, heterostructures, and surface modification. Such modifications improve the sensitivity and selectivity, reduce poisoning of the sensor materials, response, recovery time, *etc.*

ZnO is one of the first ceramic oxide NFs that is fabricated using the electrospinning process. Among much electrospun metal oxide NFs, ZnO NFs are widely used for sensing various gases such as ammonia,^[12] nitrogen dioxide,^[13,14] methane,^[15] acetone,^[16] hydrogen,^[17] hydrogen sulfide,^[18] carbon dioxide,^[19] ethanol,^[20] and carbon monoxide.^[21,22] The ZnO is advantageous over the other metal oxides for gas sensing due to its n-type semiconducting nature with a bandgap of 3.37 eV. Moreover, ZnO nanostructures can be easily fabricated using simple chemical methods, and they can be easily grown as aligned nanowires on the required substrate. ZnO is a low-cost and non-toxic material; therefore, ZnO is used in myriads of applications, such as biomedical implants, dye-sensitized solar cells, photocatalysis, photovoltaics, sensors, *etc.*

ZnO NFs are commonly used in chemo-resistive gas sensors. The sensitivity of ZnO NFs is highly dependent upon the chemisorbed oxygen species on the surface of the ZnO nanostructures. The chemisorbed oxygen accepts an electron from the conduction band of ZnO and thus increasing its resistance. During sensing, the chemisorbed oxygen on the ZnO surface reacts with the reducing gases, and the resulting electrons will be donated to the conduction band of ZnO. As a result, one can observe a reduction in resistance. The sensitivity and other sensor characteristics largely depend on the surface-active groups. The high-temperature synthesis of ZnO NFs through electrospinning favors the sensor response. The electrospun NFs have better resistive gas sensor response than rod-like short NFs and nano-powders, owing to the high surface area of the NFs and the contact-controlled effect of the fiber junctions.^[23] The barrier potential at the junction of the fibers is significantly reduced during the interaction of the adsorbed oxygen and the target gas. The vast number of nanosized grains composing the NFs increases the effective surface area of the fibers. Though there are several methods to increase the sensitivity, response time, selectivity, *etc.*, by post-processing the fibers, some techniques can be incorporated during the production stage of the NFs.

So far, a huge number of articles are available in the literature on the electrospun ZnO NFs for sensing various gases. However, a comprehensive review comprising the production processes, sensor type, mechanism of sensing, and

methods to enhance the sensitivity of ZnO NFs is not available in the literature. Therefore, this review article is intended to cover the recent advancements of electrospun ZnO NFs used for gas sensors, which helps researchers in this research area.

2. Electrospinning of nanofibers

Recently, the ZnO NFs have become an attractive material for sensors due to their physical, functional, and structural properties.^[24] NFs are used in batteries,^[25,26] fuel cells^[27] and supercapacitors^[28,29] in the energy sector; in dust filters,^[30,31] water filters,^[32,33] face masks,^[34] and photocatalysis^[35,36] in the environment sector; and in drug delivery,^[37] tissue engineering,^[38] and wound dressing in the health care sector.^[39] Fig. 1 depicts the various manufacturing methods used to synthesize ZnO NFs. Among the different NF fabrication methods, electrospinning is highly preferred because of its versatility, availability of different precursors, low cost, accessibility, and the possibility to obtain long and continuous NFs with high surface area and optical properties.^[40]

In the typical electrospinning synthesis of ZnO NFs, there are three major steps. In the first step, a spinnable viscous solution containing a polymer and zinc salt such as zinc acetate, zinc nitrate, *etc.*, is prepared in an appropriate solvent. This solution is spun as NFs using an electrospinning unit in the second step. In the third and final step, the obtained electrospun composite fibers are calcined above the degradation temperature of the polymer and the metal salt to obtain the ZnO fibers.^[41] Fig. 2(a) illustrates the schematic of an electrospinning setup. The viscous precursor solution that comes out from the needle tip of the syringe pump forms a hemispherical droplet (Fig. 2(b)) due to the repulsive Coulomb force created between surface tension and electrostatic charge. Further, under high voltage, the electrostatic force overcomes the surface tension. As a result, the droplet is stretched into a conical shape, as shown in Fig. 2(c), and it is called a “Taylor cone.” Due to the polymer stream’s high molecular cohesion, a charged liquid jet is formed and travels toward the collector (Fig. 2(d)), which is grounded. During the flight of the nanosized jet to the collector plate, it undergoes whipping, and the solvent in the jet evaporates by the end of its travel. Finally, the solvent-free jets are collected as continuous NFs onto the collector (Fig. 2(e)). Thus, formed NFs are woven in random and it is called a nonwoven NF mat. If the precursor solution is less cohesive, electrospraying occurs and results in the formation of droplets. Adding attachments such as mandrill, multiple syringes, and winding controls can produce various hybrid NFs and hollow NFs with different orientations and surface morphologies.

2.1 Parameters influence the electrospun nanofibers

Even though electrospinning is one of the easiest processes to fabricate nano- and microfibers, various parameters may significantly affect the formation of continuous and uniform NFs. These are mainly classified as (i) Solution parameters:

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Fig. 1 Nanofiber manufacturing methods: (a) Electrospinning, reproduced with permission from [42] Copyright 2019, Scientific Research Publishing Inc. (b) Self-assembly, (c) Hydrodynamic direct writing, reproduced with permission from [43] Copyright MDPI and (d) Centrifugal jet spinning, reproduced with the permission from [44] Copyright 2019, The Royal Society of Chemistry.

polymer concentration, ionic strength, solvent, conductivity, surface tension, viscosity, and molecular weight. (ii) Machine and process parameters: feed rate, needle diameter, needle to collector distance, voltage and annealing temperature, and duration of the annealing process. (iii) Ambient parameters such as temperature and humidity of the spinning chamber. The above parameters in general, affect the continuous production, physical, morphology, electrical, chemical, and structural properties of electrospun fibers, and the detailed classification is presented in Table 1. The most important parameters are discussed in the sections below.

2.1.1 Effect of voltage on the quality of the nanofiber

The voltage applied to the tip of the needle is a major component in regulating the diameter of the NFs. The solution from the syringe needle gets charged and the droplets form into the Taylor cone, as shown in Fig. 2(c). If the voltage applied is below a specific value, bead formation takes place. Many researchers have investigated the role of the voltage applied during the electrospinning process of ZnO NFs. Nonato *et al.*^[45] prepared ZnO NFs using the precursor solution containing 0.074 g/mL polyvinyl alcohol (PVA) and 0.074 g/mL zinc acetate (ZnAc₂). Electrospinning was

performed at RT using a 20 mL syringe and a 4 cm long metal needle, a needle tip-to-collector distance of 10 cm and 15 cm, feed rate of 0.0388 mL/min and 0.0194 mL/min, a voltage of 17 and 22 kV, respectively. An increase in the diameter of PVA/ZnAc₂ composite fiber was observed with the decrease in applied voltage and this was due to reduced volumetric charge density with the reduction of voltage. Additionally, the above study reveals that the voltage is not as significant as compared to flowrate. Saidin *et al.*^[46] utilized a flow rate of 0.03 mL/min, 10–20 kV voltage with a fixed distance of 16 cm to produce ZnO NFs. They observed a decrease in fiber diameter as the voltage increased and reported that it could be due to the change in electrical resistance in the working material. The continuous fibers were observed at a voltage of 14 kV with an average fiber diameter of 186 ± 62 nm. Dandan *et al.* proved a synergic relation between the concentration of the polymer, voltage, and dopant on the fiber diameter of electrospun PVA-ZnAc₂ NFs.^[47] Zahmatkesh *et al.* observed thinner NFs at voltages below 20 kV without the presence of bead formation at an optimum feed rate of 1 mL/h.^[48] Thus, as per the literature, to obtain continuous and bead-free NFs the voltage should be in the range of 15–20 kV. However, the fiber diameter depends on various parameters such as feed rate,

Fig. 2 (a) Schematic diagram of ES unit, (b) charged polymer droplet, (c) Taylor cone, (d) continuous NF formation, and (e) Nanofiber.

Table 1. Parameter influencing the physical, chemical, mechanical, and morphology characteristics of electrospun NFs.

Sl. No.	Parameters	
1	Solution parameter	Concentration → Directly proportional to the fiber diameter
		Ionic strength → Directly proportional to the charge density
		Solvent → Inversely proportional to the bead density
		Viscosity → Parabolic relation to the diameter
		Molecular weight → Increases the viscosity of the solution
2	Machine parameter	Feed rate → Directly proportional to the electric current
		Feed rate → Directly proportional to the fiber diameter
		Feed rate → Inversely related to the surface charge density
		Voltage → Inversely proportional to the surface charge density
3	Process parameter	Voltage → Inversely proportional to the fiber diameter
		Voltage → Direct influence on the bead formation
		Needle diameter → Directly proportional to the electric current
		Needle-to-collector distance → Inversely proportional to the bead formation
4	Ambient conditions	Needle-to-collector distance → Inversely proportional to the electric field strength
		Needle-to-collector distance → Inversely proportional to the fiber diameter
		Annealing temperature → Appropriate temperature to remove the solvent
4	Ambient conditions	Temperature → Inversely proportional to viscosity
		Humidity → Effect on moisture content in fiber

needle tip to collector distance, the viscosity of the spinning solution in addition to the environmental conditions.

2.1.2 Effect of solution flow rate on the nanofibers

The solution flow rate is the speed at which the charged jets disperse from the syringe needle. If the flow rate is very low, then a small quantity of solution will be ejected from the tip of the needle leading to the formation of tiny droplets. Conversely, if a large amount of solution is dispensed from the needle tip as the flow rate is increased, that may result in the formation of beads because of the non-evaporation of the solvent thus increasing fiber diameter.^[49] Nonato *et al.*^[45] examined the effects of flow rate on the electrospun ZnO NFs. They considered the flow rate from 0.0194 – 0.0388 mL/min to obtain the NFs. They observed an increase in the mean diameter of the NFs as the flow rate increased. Most of the uniform fibers were observed at a lower flow rate. At a higher flow rate, stretching of the solution jet from the needle tip was incomplete resulting in the increase of the diameter.^[50,51] Khusaini *et al.*^[51] found irregular fibers with defects and an increase in the average fiber diameter as the flow rate increased. They also observed the formation of branched NFs, ruptures, and lumps in the produced NF. The average size of ZnO NF for a flowrate of 4, 6, 8 $\mu\text{L}/\text{min}$ were 138.2, 152.3, and 173.3 nm. An ideal flow rate of 0.3-0.6 mL/h can be set to achieve a continuous flow of the solution jet and to obtain bead-free NFs.

2.1.3 Effect of needle tip-to-collector distance

The distance between the tip of the needle and the collector has been instrumental in deciding the diameter and structure of electrospun NFs. The morphology will be easily affected because the solution will evaporate during ejection until it reaches the collector if the distance is too long. If the distance is less, a wet electrospun fiber reaches the collector, leading to a beaded structure. Sara *et al.* observed that increasing the distance from 5 cm to 15 cm resulted in better distribution of the NFs.^[48] Akram *et al.* observed a decrease in the solution evaporation as the distance increased. This results in the elongation of the solution jet decreasing the fiber diameter.^[52] Therefore, to obtain smooth and uniform NFs, an optimal distance must be maintained.^[53,54]

2.1.4 Effect of polymer concentration and solution viscosity

This is one of the major aspects that determines the stability of the charged jet coming out of the needle tip. When the polymer concentration in the solution is small, the surface tension and electric field break the polymer link established to create the fiber; as a result, the fiber doesn't reach the collector.^[55] Likewise, when the concentration is increased, the viscosity increases such that the fluid becomes very thick and gets blocked in the midway of the needle tip which hampers the easy flow of the solution and increases the fiber diameter.^[56] Gazquez *et al.* studied the various factors that affect the

electrospun NFs. They agreed with other researchers that the polymer concentration and viscosity were the key factors in the electrospinning technique, which would decide the fiber diameter and the structure.^[57] Ghafari *et al.*^[24] observed a nonwoven type of structure in the spun NFs by varying the polymer concentration. They found a large number of beads for the fibers with a viscosity below 325 cP, which can be attributed to the Rayleigh instability of the fluid jet.^[58] No bead structures were observed with a viscosity higher than 325 cP.

2.1.5 Effect of solution conductivity

Solution conductivity is one of the critical parameters which affect the formation of jets in the needle tip. If the conductivity of the solution is low, there will be not enough charge present on the droplet's surface, resulting in non-formation of the fiber. Similarly, if the conductivity is increased, the charges present on the droplet's surface tend to form a Taylor cone and decrease the fiber diameter.^[59] Asli *et al.*^[60] observed thinner and bead-free NFs by increasing the solution concentration. This is due to viscosity which aids the overlapping of polymer chains and allows the electrified jet to extend continuously for fiber formation. Mansouri *et al.*^[61] observed the addition of ZnAc₂ considerably reduces the diameter of the PVDF/ZnAc₂ fibers. This is attributed to the presence of large amounts of ZnAc₂ which increases the conductivity of the solution. Sara *et al.*^[48] found a decrease in the solution conductivity when the polymer concentration exceeded 12 wt%. This resulted in the instability of the fibers. The addition of salts in the solution enhances the conductivity, which results in fewer beaded fibers. Thus, an increase in the solutions' conductivity would reduce the diameters of the fibers. If conductivity decreases, droplets of the solution at the tip of the needle cannot form the Taylor cone.

2.1.6 Influence of humidity and temperature

Ambient conditions such as temperature and humidity are influential factors in the morphology & structure of the electrospun NF.^[62] Liu *et al.*^[63] reported the decrease in the diameter of the NF as the temperature increased, while more pore formation was observed on the surface of the NF as humidity increased.^[64] Kopp *et al.*^[65] observed both temperature and humidity played a crucial part in the morphology of the electrospun NF. They found large deviations, unequal and nonwoven fiber diameters on increasing the temperature and humidity. They observed good fibers at a temperature of 25 °C and 25 % RH. Salahuddin *et al.* reported that solution concentration, voltage, feed rate, temperature, and humidity influence the electrospinning process and resulting fiber uniformity. Hence, the complex interaction of these parameters needs to be considered to design the experiment depending on the electrospun unit.^[66] Vadim *et al.*^[22] investigated the effect of humidity change in dry (0% RH) and humid conditions (60% RH). They found that increasing the humidity at dry conditions causes the pure ZnO NF sensor response to vanishing completely in the

temperature range of 100–450 °C. Likewise, the sensor response of ZnO/Pd NFs changed marginally when air humidity increased up to RH = 60%.

The NFs produced by electrospinning then undergo calcination in an oven for a time period at elevated temperature to remove all the impurities and solvents. It is important to note that the calcination temperature, heating rate, and dwell period at high temperature does affect the structural characteristics of the ZnO NFs. Hence, preparation parameters of the electrospinning technique play a vital role in getting desired NFs for a specific application.

3. Gas/vapor sensing

3.1 Types of gas/vapor sensors

The gas/vapor sensors are generally classified as acoustic wave, resistive, photoelectric, and optical gas sensors. The concept of acoustic sensors is adopted from Sauerbrey's quartz crystal microbalance, which was first used to detect organic vapor.^[67] The application of surface acoustic wave (SAW) technology can be extended for the detection of gases as well.^[68,69] The application of SAW sensors is flexible, and it is widely used for the detection of various gases,^[70–72] environmental changes such as pressure,^[73] humidity,^[74] UV radiation,^[75] *etc.*, besides the chemical and biological sensing applications.^[76,77] Sensitivity, reversibility, less reaction and desorption time, and high selectivity are the important performance characteristics of SAW sensors.^[78] Most SAW resonators have sensitive films which are exposed to the target gas, and the sheet conductivity, elastic modulus, and weight of films will vary as the film interacts with the target gases. Such variations significantly impact the propagation of acoustic waves on the surfaces of SAW resonators. The effect of target gas on the propagation of acoustic waves is the response of SAW-based sensors and it is different for dissimilar molecules. A photoelectric sensor detects the distance traveled by light in the absence or presence of target molecules by the light transmitter and receiver. They are frequently utilized in the manufacturing industry. Photoelectric technology has progressed to the point where a sensor can identify a target with a diameter of less than 1 mm. The principle of surface photovoltaic (SPV) response has been found in many oxide semiconductors such as ZnO, SnO₂, TiO₂, WO₃, *etc.*^[79–83] When a semiconductor is excited by light with photon energy larger than or equal to the bandgap, the surface potential of the semiconductor changes. When the detected gas gets adsorbed on the surface of the semiconductor, there will be a transfer of electric charges between the adsorbed gas and the semiconductor. This causes a change in the value of the surface dark potential leading to the difference in the SPV. The photoelectric gas sensor has the advantage of being able to operate at RT.

Optical sensors are the type of sensors that are lightweight, tiny in size, and have high sensitivity.^[84] Optical sensors generally exhibit improved sensitivity and selectivity at RT. The sensing technique in optical sensors is based on cladding

alteration, which involves removing a tiny portion of the optical fiber's clad and coating a side polished area with the active gas sensing material.^[85,86] Due to the gas interaction, the sensing medium's refractive index varies, leading to a change in the intensity of light traveling through the fiber. The leakage of light by the modification of the cladding resulting in the change of wavelength and refractive index.^[87] In this type of sensor, the nanomaterials have drawn a lot of interest because of their high gas sensitivity which is caused by their strong surface activity.^[88]

Resistive sensors are the most prevalent type of sensors that are extensively used in daily life. A resistive sensor is an electromechanical device or transducer that converts physical or mechanical changes such as displacement, stress, or strain into an electrical signal that can be monitored after conditioning. Wheatstone bridges are frequently used in conjunction with resistive sensors. It is also used for measurements that produce an output in the form of resistance, for example, piezoresistive resistance changes with a change in pressure. In the case of light-dependent resistors, the resistance changes based on the change in light intensity and so on.

3.2 Gas-sensing Mechanism of resistive type ZnO based gas sensors

Based on the sensing technique, gas sensors are classified as acoustic wave, resistive, photoelectric, and optical sensors.^[89] In this paper, we restrict the discussion to resistive type gas sensors due to their wide acceptance as reliable sensors. The oxide semiconductor gas sensors exposed to toxic and explosive gas environments show a drastic change in their resistance. In n-type semiconductor type resistive sensors, the initial resistance is the highest under normal atmospheric conditions due to the adsorption of negatively charged oxygen which forms an electron depletion layer on the surface. The reducing gases present in the atmosphere or the test chamber get oxidized by the reaction with negatively charged surface oxygen, and electrons are injected into the semiconducting core, which ultimately decreases the sensor resistance.^[90] Fig. 3 depicts the resistive type of gas sensing mechanism and a representative testing arrangement.

When the sensor is in contact with oxidizing gases such as NO₂, O₂, CO₂, the thickness of the potential barriers increases, and the difference in thickness regulates the resistance of the sensor. This enhances the sensor response because of the extraction of electrons from the surface of the gas sensors. Likewise, when the sensors are in a reducing medium such as C₆H₆, CO, NH₃, CH₄, or NO, as a result of the interaction between the reducing gas and adsorbed oxygen ions, the liberated electrons return to the surface of the sensor and reduce the thickness of the potential barriers, enhancing sensor response. Fig. 4 illustrates the schematic sensing mechanism in the (a) ambient air, (b) oxidizing gas, and (c) reducing gases.

Fig. 3 The schematic block diagram of (a) gas sensing mechanism and (b) gas sensing setup.

3.3 Characteristics of resistive gas sensors

The essential characteristics of an exemplary gas sensor are depicted in Fig. 5. The gas sensor sensitivity for the particular test gas depends on the concentration of gas, operating temperature (T_0), and electronic and structural characteristics of the gas sensor material.^[91] The researcher's challenging task is to develop gas sensors that give high sensitivity and operate near RT to sense a small gas concentration in the test chamber. The gas sensitivity can be calculated using the equation, $\text{Sensitivity} = \frac{C_v}{I_C - I_0}$, I_C is the sensor output current when the test gas concentration is C_v (ppm), and I_0 is the sensor output current without any test gas. The sensors response can be defined mathematically as, $S = \frac{R_0}{R_{\text{gas}}} - 1$, where R_0 is the sensor resistance in clean air, and R_{gas} is the sensor resistance

in the presence of reducing gases. For oxidizing gases, the sensor response can be computed as $S = \frac{R_{\text{gas}}}{R_0} - 1$.^[92] If the resistive type of gas sensors can produce reproducible results over a specific period, then one can say that the sensor is stable. The stability of the sensor includes the retention of sensitivity, selectivity, response, and recovery time. The stability of the gas sensor is the most challenging issue in metal oxide sensors. Nanostructured oxide materials may easily degrade due to their high reactivity, which ultimately leads to stability problems in metal oxide sensors. The stability of the metal oxide sensors can be improved by doping metal particles, carbon nanotubes, and the synthesis of mixed oxides.^[93,94] Additionally, the post-processing, such as calcination, annealing, and reducing the working temperature of sensors improve the stability.

Fig. 4 Gas sensing mechanism of ZnO NFs in (a) air, (b) oxidizing, and (c) reducing gases. Reproduced with the permission from [95] Copyright MDPI.

- Electronic structures of metal oxide
- Band gap
- Electrical conductivity
- Surface area per unit volume
- Porous
- Grain size of crystalline ZnO on nanofiber
- Size of nanofiber

Fig. 5 Characteristics of gas sensors.

4. Methods to improve the ZnO nanofiber resistive gas sensor characteristics

The sensing characteristics of electrospun NFs can be effectively improved through different approaches. The common approaches such as tailoring the structure, doping, functionalization of the NFs, adding conductive materials, composite formation, and electron and ion beam irradiation are discussed in the subsequent sessions. In addition, there are numerous ways of enhancing the sensitivity of the fabricated sensor. This includes selecting an appropriate operating temperature based on the analyte and using sensor arrays.^[96]

4.1 Effect of structure on the sensitivity of resistive sensors

The sensitivity of nanostructured materials for gas sensing is often determined by particle size, morphology, crystallinity, *etc.* The electrospun ZnO NFs are composed of a large number of grains with sizes ranging from 20 to 80 nm, and they are polycrystalline.^[97] The grain size and crystallinity of electrospun ZnO NFs vary with the calcination temperature. Katoch *et al.* found that both crystallinity and grain size influence the sensitivity of ZnO NFs toward CO. If the crystallinity is too low, the sensitivity is poor, and it increases with the crystallinity. Similarly, as the grain size increases, the sensor response improves up to a certain grain size but decreases thereafter. The calcination temperature influences both crystallinity and grain size. As the calcination temperature increases both crystallinity and grain size increase. At high calcination temperatures, the effect of grain growth is more significant. When comparing the sensor response, the large grain size obtained at high calcination temperatures reduces the sensor response.^[97] Park *et al.*^[98] observed the presence of nanograins in the ZnO NF calcinated at 600 °C (Fig. 6a). They found the as spun NFs exhibit smooth surface with a uniform diameters. During calcination, the NFs show slight shrinkage with a grainy structure. As the temperature is increased to 600 °C the NFs consist of much larger grains indicating the presence of grain growth (Figs. 6(b-c)). Increasing the duration of calcination enhances grain growth in the ZnO NF (Figs. 6(d-e)). Therefore, one has to optimize both crystallinity and grain size to achieve a maximum sensor

response.

Creating hollow NFs increases the surface area of the NFs. Interestingly, variations in the hole diameter of ZnO hNFs affect the sensor response. hNFs with small hole diameters exhibit more sensitivity than the ones with large hole diameters towards reducing and oxidizing gases. The improved response of hNFs with small hole diameters is due to the increased surface area of those fibers.^[99] Post-processing of electrospun ZnO NFs by hydrothermal method results in the formation of nanorods on the NFs creating a brush-like appearance on the fibers. The brush-like hierarchical growths on the NFs tend to increase the surface area of the NFs tremendously. Comparing the sensitivity of spun ZnO NFs with brush-like ZnO NFs reveals a remarkable increase in the sensor response using the latter towards toluene. The increase in surface area in brush-like NFs leads to an absorption of more target gas and offers additional active sites for interaction with the analyte. The more absorbed gas interacts with adsorbed oxygen, releasing trapped electrons to the conduction band of ZnO, resulting in a reduction of the resistance, and one can record more response and sensitivity.^[100] The brush-like ZnO NFs are more sensitive to toluene than CO, which may be attributed to the preferentially grown 1-D nanorods that have more affinity to toluene than CO.

Sanger *et al.*^[101] investigated the sensitivity of the NO₂ gas sensors at RT by fabricating aluminum-doped ZnO by the electrospinning method. The hexagonal wurtzite structure of ZnO was observed with the corresponding crystalline plane orientation of (101). They observed the formation of hollow NFs having an average diameter of 200 nm. The transmittance spectra of the filled fibers are about 80% in the visible range, and that of NFs is close to 90%. Due to the presence of Al₂O₃ in the filled fibers, a decrease in transmittance is observed. This was due to the Burstein Moss effect wherein the fermi level of Al-doped ZnO fibers penetrates the conduction band in the presence of UV light and fills it with electron charge carriers resulting in the decrease of transmittance. The aluminium-doped NFs have a high surface-to-volume ratio, making them

Fig. 6 Field Emission Scanning Electron Microscope (FESEM) images of (a) The presence of nanograins in the fiber, (b-c) ZnO NFs calcined at 500 °C and 600 °C temperature, (d-e) ZnO NF calcined at 600 °C for 6 hours and 8 hours. Reproduced with permission from [98] Copyright 2009 The American Ceramic Society.

a good gas detecting material at RT for 0.5 ppm NO₂ gas. The sensitivity of the aluminum-doped NFs recorded as three times higher than that of the undoped ZnO fibers. Figs. 7(a) and (b) depict the image of hollow and filled NF. The response and recovery time of the sensor to 0.5 ppm of NO₂ gas is shown in Fig. 7(c). Figs. 7(d-f) summarizes the active gas sensing distribution in the hollow and filled NFs. The gas interaction mainly depends on the energetic gas molecules, the number of active sites, and the collision frequency. At higher temperatures, energetic molecules adsorb at the surface quite easily, and at lower temperatures, it is slow to react. The collision frequency is governed by the shape of the NF and the temperature. The collision frequency inside the hollow doped ZnO fibers core is eight times higher than the outer surface. This attributes to the longer path and more space available on the hollow core as the gas molecules get trapped, thus decreasing the barrier height. This results in higher sensitivity.

4.2 Enhancing the sensitivity by doping

Doping is the process of adding a dopant to the prepared oxide, which can be done by impregnation and chemical mixing. Chemical mixing is the typical approach used in the fabrication of doped ZnO NFs. Adding additives to ZnO creates new active centers and helps in modifying the crystal structure and properties.

Transition metals like Ni, Co, Fe, and Cu are frequently used as dopants in ZnO nanomaterials.^[102–106] These dopants tend to enhance active sites, change ZnO resistance, lower the sensor operating temperature, improve the selectivity and stability of the sensor, and accelerate reaction to the target gases.^[107,108] The surface morphology of ZnO nanostructures

alters as metal dopants are introduced.^[109] The growth of crystallites is often restricted by the boundary interaction between the dopant and the host material, resulting in smaller grain sizes in doped ZnO NFs than in pristine ZnO.^[110]

Platonov *et al.*^[111] studied CO sensing characteristics of as-spun and Pd-doped ZnO NFs in the dry and humid air. The pristine ZnO sensor loses its sensitivity to CO under moist conditions (RH = 60%) while Pd doped ZnO sensor records a higher sensor response. This is due to the Schottky sensitization effect where there is a decrease in the potential barrier at the surface of ZnO, which reduces the formation of PdOx clusters at the ZnO/Pd interface upon exposing the sensor to CO gas. To investigate the H₂ sensing characteristics of NFs Lee *et al.*^[112] synthesized Co₃O₄ doped ZnO NFs with different amounts of cobalt oxide via electrospinning. Compared to other concentrations, the maximum response to 10 ppm of H₂ gas is recorded for 0.05 wt% Co₃O₄ at 300 °C. When the H₂ gas is exposed, the resistance in the ZnO grain boundaries decreases in the presence of Co₃O₄, resulting in the maximum reaction. Liu *et al.*^[113] investigated the influence of metal ion doping on the photocatalytic performance of pristine and Fe (0.5, 1, 1.5, 2 wt%) doped ZnO NFs. The NF's smaller diameter helps to increase the surface area of the NF, attributes to the good photocatalytic capacity. The optical band gap is reduced when the Fe ions are doped, thereby increasing the photocatalytic activity. The 1.5wt% Fe-doped ZnO NFs have the best photocatalytic performance of all the Fe concentrations because they have the most dopant ions in the ZnO sample. The bandgap is reduced from 3.24 to 3.16 eV as a result of this.

Fig. 7 SEM micrographs of (a) hollow NF, (b) filled NF, (c) response and recovery time of hollow NF to 0.5ppm NO₂ gas at RT, the interaction of gas molecules with (d) outer surface of the filled fiber, (e) outer and inner surface, and (f) gas sensing mechanism. Reproduced with permission from [101] Copyright 2018, WILEY-VCH Verlag GmbH & Co. KGaA, Weinheim.

They concluded that samples with smaller optical band gaps have a wider light absorption range, which contributes to improved photocatalytic activity. Baylan *et al.*^[114] explored ZnO NFs photocatalytic characteristics by doping manganese (0-1 wt%) using the ES process. Since the ionic radius of Zn is less than that of Mn, the X-ray powder diffraction (XRD) analysis shows that the inclusion of Mn reduces the crystallite size of ZnO. According to the X-ray photon spectroscopy (XPS) results, Mn doping occurs in the ZnO crystal in the ionic states of Mn²⁺ and Mn⁴⁺, resulting in the formation of new energy levels within the ZnO bandgap. The oxygen vacancy is the major contributor in 0.5 wt% Mn-doped ZnO fibers. This results in the formation of tensile stress due to the increase in the defect concentration. The doping of Mn increases the degradation activity of the NFs. 100% degradation of methylene blue degradation is achieved at 90 minutes under UV light and 35% at 100 minutes under visible light for 0.5 wt% Mn-doped NF. This helps in enhancing the surface area of the NF. The 0.5 wt% Mn doped NFs display high stability and durability in the degradation tests, checked after ten cycles.

4.3 Functionalization of noble metal nanoparticles

The addition of noble metals helps to alter the metal oxide's surface which enhances the sensor sensitivity and selectivity. In the sensing mechanism, various noble metals such as Au, Pd, and Pt act as catalysts or sensitizers.^[115,116] During the gas sensing process, they establish a heterogeneous interface with the host metal oxide, which sensitizes/changes the depth of the electron barrier and modifies the kinetics of electron transfer between metal and oxygen. Sensitization is generally caused by two mechanisms, electronic and chemical sensitization. Electronic sensitization is generated by electronic interfacial distribution, in which electrons are removed from metal oxides and transferred to noble metals due to a work function difference.^[117] This increases the height of the depletion layer in the oxide. Since the potential barrier height is larger than undoped grains, the noble metal-doped sensor will be more sensitive to the environment. The chemical sensitization of noble metals is known as the spillover effect.^[118-121] It is caused by the interfacial transport of atoms.^[122] In this effect, the deposited noble metals create

Fig. 8 (a-b) FESEM micrographs of pure and Pd/ZnO NFs, (c) Resistance values at dry and humid conditions (d) Sensor response when exposed to carbon monoxide gas at different temperatures. Reproduced with permission from [22] Copyright MDPI.

more active sites for oxygen adsorption and the target gases, which facilitates molecule dissociation due to the high catalytic nature of the noble metals. Ionic oxygen species adsorbed on metal oxide nanoparticles will permeate the surface region at noble metal-supported interfaces in the presence of air. These metal nanoparticles activate the spillover of detected gases, which are then oxidized by the ionic oxygen species during the sensing process. This leads to an increase in the transfer of electrons onto the metal oxide. Xuan *et al.*^[123] prepared Au-doped ZnO NFs with an average diameter of 100 ± 20 nm. They observed uniformly distributed Au nanoparticles with the face-centered cubic structure on the surface of ZnO NF with a hexagonal wurtzite structure. The nanofibrous structure decorated with Au nanoparticles enhances the NF's electrical conductivity. The molar ratio of Au/Zn plays an essential part in improving the sensor response and tuning the NF structure. The maximum response of the Au/ZnO sensor is recorded for an atomic ratio of 0.24 for the H_2O_2 gas. They concluded that the Au nanoparticle's catalytic nature increases the sensor response compared to the pristine ZnO sensor.

Vadim *et al.*^[122] investigated the sensing properties of carbon monoxide (CO) in dry and humid conditions by preparing pure and Pd-doped ZnO NFs. A spongy structure of pure ZnO with a fiber diameter of 500-1000 nm is observed, while the introduction of Pd particles decreases the diameter of the NFs (150-200 nm) (Figs. 8(a-b)). The characteristics of the pristine and Pd-doped ZnO sensors to CO gas are studied at dry (0% RH) and humid (60% RH) conditions. The formation of a potential barrier at the ZnO/Pd interface is

observed, resulting in the increase of resistance (Fig. 8c). The higher work function of the Pd (6.04 eV) than ZnO (4.6 eV) contributes to the formation of the potential barrier. The maximum sensor response is observed at a temperature of 300 °C on the dry conditions for the ZnO/Pd sensor (Fig. 8d). This is due to the electronic sensitization effect, which decreases the potential barrier thus enhancing the sensor response.

Sun *et al.*^[124] investigated the sensing characteristics of pristine ZnO, Co_3O_4/ZnO , and Pd/ZnO and Pd/ Co_3O_4/ZnO NFs fabricated by the electrospinning technique. A decrease in the fiber diameter is observed when dopants such as Pd and Co_3O_4 are added. Pure ZnO has an average diameter of 172 ± 22 nm, while Co_3O_4/ZnO , Pd/ZnO, and Pd/ Co_3O_4/ZnO NFs have diameters of 92.1 ± 15 nm, 113 ± 8.3 nm, and 85.9 ± 15.8 nm, respectively. The mesoporous nature of the NF is observed from the pore size distribution, which favors the gas sensor.^[125] The Pd/ Co_3O_4/ZnO NFs exhibit the highest specific surface area ($12.66 \text{ m}^2 \cdot \text{g}^{-1}$) among all NFs. The Pd/ Co_3O_4/ZnO sensor records the highest response (59 at 240 °C for 200 ppm of ethanol) among sample sensors compared with different volatile organic components. This high response is attributed to Pd's electronic and chemical sensitizations as well as the formation of the p-n junction at the interface of Co_3O_4 and ZnO.

Kanimozhi *et al.*^[126] fabricated pure and Ag (5, 10%) doped ZnO NFs using the electrospinning technique. The structural results confirm the pristine and Ag-doped ZnO NF exhibit the preferred wurtzite structure. The gradual decrease in the bandgap on the addition of Ag is observed, allowing the Ag–

ZnO NFs to absorb a wide range of visible light. A bandgap of 3.28 eV is found for pristine ZnO and reduced to 2.93 eV for the 10% Ag-ZnO NFs. The fabricated samples are used as photoanode for dye-synthesized solar cells by pre-treating the NF with TiCl_4 . The Ag-doped ZnO NF records higher power conversion efficiency of 5%, and the pristine ZnO NF shows a value of 3.19% when used as a photoanode. The high-power conversion efficiency of the Ag-doped ZnO NF can be attributed to the surface plasmon resonance effect of the silver nanoparticles distributed on the ZnO NFs' surface.

4.4 Inclusion of carbon-based nanomaterials

The field of carbon-based NFs has been gaining more importance in technological development because of their mechanical strength, chemical and physical properties, and electrical conductivity.^[127,128] Surface modification of carbon-based NFs can be used to fabricate hybrid carbon NFs-based nanomaterials that are commonly used in biomedicine,^[129] tissue engineering,^[130] nanodevices,^[127] sensors,^[131,132] energy,^[133] and environmental science.^[134] Many researchers have reviewed the synthesis and application of carbon NFs.^[135–137] Zhang *et al.* illustrated various strategies for generating carbon-based NFs with an electrospinning technique and outlined their applications in various fields.^[135] Feng *et al.* investigated the synthesis, characteristics, and applications of carbon-based NFs. The various applications used in electrical devices, batteries, and supercapacitors were studied.^[136] Zhang *et al.* summarized the recent developments in electrospun fiber synthesis and electrochemical energy storage applications.^[137,138] Zhang *et al.* studied an H_2S sensor based on ZnO/CNFs nanocomposites, which demonstrated a linear response in the range of 50–102 ppm of H_2S gas.^[139] One of the most often used materials to improve sensor activity is graphene. Because of its high electron mobility, chemically derived graphene is a susceptible chemical sensor.^[140–142] Graphene-doped sensors can detect individual gas molecules and have superior sensing behavior because they are shallow noise materials.^[143,144] The reduced graphene oxide (rGO) decorated with oxygen functional group provides more adsorption sites, which helps to improve the sensor sensitivity.^[145–147]

Salehi *et al.*^[148] studied the gas sensing mechanism of acetone gas by doping reduced graphene oxide (rGO) to the zinc acetate solution by electrospinning technique. Different varying zinc acetate and graphene ratios are selected to obtain the optimal concentration for a high response. The clear dispersion of reduced graphene in the ZnO fibers is observed, and the diameter of the calcined NFs is in the range of 190–480 nm. High sensitivity to the acetone gas is found as the weight fraction of zinc acetate increases. The doping of the reduced graphene oxide increases the sensitivity of the sensor from 2.3 to 4 with a decrease in operating temperature.

Abideen *et al.*^[149] fabricated rGO-loaded ZnO NFs functionalized by Au and Pd nanoparticles via electrospinning. The Au and Pd-functionalized sensor exhibit a superior gas

response to CO and C_6H_6 gases at 400 °C. The electron depletion layer in ZnO NFs is broader due to the formation of ZnO/rGO heterojunctions. An increase in conductivity is observed when the sensor is exposed to reducing gases. This is due to the reaction with the oxygen species which liberates electrons and contributes to the higher response. Since rGO has a different work function and higher surface area than ZnO, it enhances the sensing response. In the case of electronic sensitization, the oxidized Pd (PdO) is reduced in the presence of C_6H_6 after taking electrons from ZnO. As a result, the electrons return to the relaxed space charge layer of ZnO. Au and Pd nanoparticles catalyze the chemical sensitization process. The incoming oxygen species bind to the surface of Au or Pd, causing oxygen to dissociate and spill over into nearby ZnO. This results in the widening of the depletion layers, which serve as a resistance modulator during the exposure of the target gases. In addition to this, the formation of Schottky barriers is observed between the Au/ZnO and Pd/ZnO interfaces. The flow of electrons from the ZnO to the metals decreases the electron depletion layer's width resulting in a significant change in the resistance of the gas sensor.

4.5 Role of composite fibers

Composite nanofibers are a novel, distinctive, and adaptable class of materials that have recently gained attention in various disciplines owing to their excellent structural and tunable functional properties. These nanocomposites have superior mechanical properties, and excellent thermal, electrical, optical, and other properties, generally at low-reinforcement volume fractions.^[150,151] The main qualities for such reinforcement effects are that (1) nano-reinforcements have far better properties than the reinforcing materials now in use, and (2) their surface area to volume ratio is exceptionally high, allowing for more interfacial interaction with the matrix. Composite NFs with their tunable mechanical and biodegradable materials have potential applications in various areas such as drug delivery, tissue engineering, stem cells, cancer therapy, and wound healing. Hung *et al.*^[152] investigated the sensing characteristics of H_2S gas by fabricating ZnO-SnO₂ fibers doped with Au crystals using electrospinning. They observed the porous structured NFs with an average diameter of 120 nm for 0.05 wt% of Au doping. The sensor exhibits high resistance when Au is doped. This is attributed to the formation of heterojunction between Au crystals and the NF. When 0.005 wt% Au doped sensor is exposed to H_2S gas, an increase in the sensor response is observed compared to the pristine sample. This is because of the synergetic impact of the heterojunction formed between ZnO and SnO₂, and the Schottky junctions between Au and metal oxide help to improve the sensing capability of H_2S gas. The maximum response is recorded for one ppm of the gas at 350 °C. They observed the presence of nanograins tends to create nanogaps in the NFs thus enhancing the response. The p-n heterojunctions play a major role in improving the gas-sensing properties of NF sensors by speeding up charge transfer.^[153]

Sabzehmeidani *et al.*^[154] synthesized different molar ratios (0, 0.2, 0.5, 0.8, and 1) Zn/Ni NFs by electrospinning and calcined at different temperatures. They observed the polymers completely decompose at a temperature of 550 °C, and the average fiber diameter is found to be 150 ± 50 nm. The wurtzite and cubic structure of ZnO & NiO is observed with the corresponding crystalline plane orientation of (002) and (111). The fabricated NFs is used as a photocatalyst to degrade a mixture of dyes (Rhodamine B and Methylene blue) in water under UV irradiation. The doping of NiO forms a p-n junction barrier which enhances the photolytic activity due to the generation of electrons and holes formed by carriers, leading to the degradation of the dyes in solution. The highest photocatalytic activity calcined at 550 °C is (ZnO_{0.8}NiO_{0.2}) composite NFs. The maximum photodegradation of RDB and MB for composite NFs (ZnO_{0.8}NiO_{0.2}) recorded is 59.41% and 65.43%, respectively.

Jatoi *et al.*^[155] investigated the antibacterial properties of 5 wt% and 10 wt% (CA/ZnO/Ag) composite NFs produced by electrospinning. They observed the hexagonal wurtzite structure of ZnO with their preferred orientation (101) plane. Similarly, the face of the Ag-ZnO exhibits a centered cubic structure with their preferred plane (111). Bead-free samples are formed and the addition of Ag nanoparticles (NPs) decreases the diameter of the NFs. Dopamine hydrochloride (Dopa) is used as a reducing agent to immobilize Ag NP's on ZnO nanoparticles. Dopa's strong adhesive qualities cause silver ions to bind to ZnO nanoparticle surfaces, and its metal ion lowering properties result in Ag NP's. The antibacterial investigation confirms the growth inhibition of *E. coli* (gram-negative bacteria) and *S. aureus* (gram-positive bacteria) strains on agar plates and liquid mediums. The generation of reactive oxygen species enhances the bacteriostatic and bactericidal performances of the CA/ZnO/AgNPs NFs. The 10 wt% (CA/ZnO/Ag) nanocomposite shows excellent cell viability against *E. coli* and *S. aureus* as compared to that of 5 wt% nanocomposites.

4.6 High energy irradiation

In electron beam irradiation, a sample is bombarded with high-energy electrons, which results in a cascade of these electrons traveling through the target material. The E-beam technique uses high-energy electrons as a source of radiation. The electrons are concentrated on a scan horn of a specific size and scanned to form a curtain of electrons in a sweeping motion. After that, the product is moved past the scan curtain at a precise and controlled speed.

Kim *et al.*^[156] reported the formation of ZnO nanograins in the nonirradiated ZnO NFs having an average diameter of 120 nm. When the NFs are bombarded at 50, 100, and 150 kGy doses, long and continuous ZnO NFs with a large surface area are observed. The peaks corresponding to crystalline ZnO are identified in the XRD patterns with their full width at half-maximum (FWHM) values found to be maximum for the 150 kGy e-beam-irradiation. This confirms that 150 kGy beam

irradiation induces structural defects resulting in crystalline degradation. As the irradiation dose increases, the atomic concentration of oxygen decreases, implying an increase in oxygen vacancies. The non-irradiated ZnO NF sample has a lower surface area than the irradiated ones. The formation of structural defects causes an increase in the surface area of e-beam irradiated NFs. The non-irradiated sensor increases the sensor response with increasing gas concentration at different temperatures. The maximum response is recorded for 0.1 ppm H₂ gas, and the response increases with the temperature, reaching an optimum value at 350 °C. Further increase in temperature the metallic Zn transforms to a liquid phase at 400 °C, disabling the sensing behavior of ZnO. Similarly, with the rise in irradiation dose, the sensor response increases, and the maximum response is observed at 50 kGy doses for 10 ppm H₂ gas. The addition of noble metals to the metal oxide structures and the e-beam irradiation enhances the response of the sensor. Kim *et al.* observed that the depletion layer thickness of the pristine ZnO NF is determined by removing ZnO surface electrons by oxygen species in the air. With the addition of palladium nanoparticles, a ZnO/Pd heterojunction is formed, and the Pd absorbs more electrons, resulting in a thicker depletion layer on the exposed surfaces of the ZnO sensor. The combination of metallization effects in ZnO, Pd's superior catalytic activity, and the formation of defects due to e-beam irradiation result in a high response.^[157]

Table 2 summarizes the gas sensor response of different types of ZnO nanostructures doped with different organic and inorganic dopants and operating at different temperatures. It can be observed from the table that most of the doping to various types of nanostructures enhances the response of the sensor at lower temperatures.

5. Selectivity of ZnO gas sensors

Gas sensors are used to detect different types of gases in various science and engineering sectors as depicted in Fig. 9. In this paper, we restrict our discussion to the selectivity of the doped ZnO NFs used for sensing acetone, ethanol, ammonia, hydrogen, and hydrogen sulfide. The performance of doped ZnO NF to detect the above-said gases is consolidated in Table 3.

5.1 Acetone gas sensors

According to literature reports, due to doping with various elements, ZnO NFs experience changes in morphology, and electronic and structural properties besides the respective sensor performance. Yuzhen *et al.*^[158] reported that doping Mn in ZnO NFs increases the surface area, in addition to the increased surface roughness and size of the NF. They observed that in doping of Mn²⁺ molecules, the surface activity of the ZnO NFs is enhanced since manganese has lower ionization energy than ZnO. At low temperatures, the reaction rate between the acetone and the adsorbed oxygen molecules will be less, resulting in higher activation energy of the ZnO surface. With

Table 2. Parameter influencing the physical, chemical, mechanical, and morphology characteristics of electrospun NFs.

Type of Structures	Dopants	Target Gas	Concentration (ppm)	Response	Response/Recovery time (s)	Temperature (°C)	Ref.
Nanorod & nanoflower	Cetyltrimethylammonium bromide (CTAB)	NO _x	0.74	29	-	RT (25)	[159]
	Citric acid monohydrate (C ₆ H ₈ O ₇ ·H ₂ O)	Ethanol	20	18	31/310	250	[160]
Nanowire	Titanium oxide (TiO ₂)	Nitrogen dioxide	5	7	65/98	200	[161]
	(3-aminopropyl) triethoxysilane (APTES)		1	160	-	RT	[162]
	Nickel oxide (NiO)	n-butanol	100	106	4/6	240	[163]
Nanocomposite	Graphene	Carbon monoxide	90	41.08	280/45	RT (27)	[164]
	Iron (Fe)	Ethanol	100	319	39/29	320	[165]
Nanoparticles	Copper oxide (CuO)	Nitrogen dioxide	100	4.1	60/25	250	[153]
ES	Copper oxide (CuO)	Hydrogen sulfide	50	31.172	105/152	180	[166]
	Zirconia (ZrO ₂)	Butanol	100	107	20/40	245	[167]
	Palladium (Pd)	Hydrogen	50	2.514	336/294	250	[168]
Nanosheets	Platinum (Pt)	Carbon monoxide	50	3.57	6/19	180	[169]

the increase in temperature, the rate of absorption equals the rate of desorption as the chemisorption reaches the highest response with the optimal temperature. They observed a decrease in resistance as the concentration of acetone increased. 2.0 wt% Mn-doped ZnO NFs sensors are found to be the most selective and dependable to acetone. Li *et al.* [170] also found an increased surface area beside a change in electronic and structural properties of ZnO hollow NF after Ce doping. When the sensor is exposed to atmospheric air, oxygen molecules get adsorbed on the ZnO surface, and extract electrons from the conduction band of ZnO, resulting in the formation of O₂⁻ and O⁻. This results in creating a depletion layer around the surface of ZnO, thereby increasing the resistance. When the adsorbed oxygen reacts with the acetone gas, it releases electrons back to the conduction band, thus reducing the thickness of the depletion layer and enhancing the sensing properties. The large surface area of the hollow NF structure facilitates the easy transport of electrons adsorbed by the oxygen molecules and increases gas sensing performance. Ce-ZnO NFs exhibit long-term stability and a more significant response towards acetone sensing than other gases. Xu *et al.* [171] observed and reported thick-closely connected and small-loosely connected nanograins on pure ZnO and La-ZnO NF, respectively. They noticed a bead-like morphology with a rough surface on 1 wt% La-doped ZnO NFs and the sensing response of these NFs increased substantially with an increase in temperature up to 340 °C and decreased with a further rise in the temperature. The improved response was attributed to the degradation of crystallinity at higher temperatures, which adversely affected the sensing response. They concluded that the 1 wt% La-ZnO sensor records the highest response for

acetone gas compared to other target gases. From the above studies, it has been observed that the Ce-ZnO hollow NFs exhibit the highest response for detecting C₃H₆O gas at lower temperatures even though the diameter of the fiber is large. This response is attributed to the higher surface area per unit volume of the hollow structured NFs, and the porous and surface roughness. 1 wt% La-ZnO fibers show a good response at low gas concentrations. It is found that the Mn and La-doped ZnO NF are smaller in size compared to Ce-ZnO NFs. Hence, the further reduction of the size of Ce-ZnO NF may improve further sensitivity for C₃H₆O gas.

5.2 Ethanol gas sensors

Sun *et al.* [172] found the size of Er-ZnO NF decreases with the increased concentration of Er. Both pure and doped NFs exhibit an improved response with an increase in temperature up to 240 °C, and further temperature rise decreases the response. Good repeatability, long-term stability, and a greater response of 1 wt% Er-ZnO NF are reported for sensing ethanol gas. Large lattice distortion was attributed to the maximum response because of the adsorption of oxygen molecules and further ionization at the NF surface. This change leads to more electron capture from the ZnO conduction band leading to an increase in the depletion layer thickness. However, pure ZnO NF exhibits good response time and recovery time (5 s and 2 s) compared to 1 wt% Er-ZnO NF (12 s and 3 s). Lai *et al.* [173] investigated Au-sputtered SnO₂-ZnO electrospun NFs, and reported decreased size, rough surface, porous structure, decreased absorption, and increased optical transmission due to an increase in calcination temperature (> 550 °C). They noticed that the heterojunction of the SnO₂-ZnO depletion

Fig. 9 Different applications of gas sensors.

Table 3. Performance of doped ZnO NF to detect various gases.

Gas type	Dopant	Concentration (Wt%)	d (cm)	v (kV)	t _c (°C)	t (hr.)	d _f (nm)	r	Gas Con. (ppm)	t _o (°C)	Ref.
C ₃ H ₆ O	Mn	1-2	25	15	580	2.5	60-90	262.5	1200	340	[169]
	Ce	1.7	15	13.4	600	1.5	197-316	364.3	1000	260	[170]
	La	0.12-2.64	23	13	650	4	60-80	64	200	340	[171]
	Er	0.5-2	12	7	600	2	200	37.3	200	240	[172]
C ₂ H ₅ OH	SnO ₂	1-2	-	17	550	2	40-260	42	100	300	[173]
	Ag	1-5	20	15	600	2	100	148	100	320	[174]
	NiO	1	-	20	500	1	450	65	250	RT	[175]
NH ₃	CoNiO ₂	0.2	15	20	400	2	60-150	240	100	220	[176]
	PSS	6-50	15	20	500	3	215	17	100	RT	[177]
	-	-	20	15	600	0.5	-	109.1	10	350	[178]
H ₂	NiO	0.03-0.15	20	15	600	2	125-200	60	10	200	[179]
	Co ₃ O ₄	0.03-0.15	20	15	600	2	100	133	10	300	[112]
	CuO	0.03-0.07	20	15	600	0.5-48	-	4489	10	150	[180]
H ₂ S	SnO ₂	0.7	15	25	600	3	-	66.23	50	250	[181]
	Ag	1-5	20	15	600	2	100	298	10	120	[174]

d- Needle tip to collector distance; v- Voltage; t_c- Calcination temperature; t – Time duration; d_f-Diameter of the fiber; r- Sensor response; t_o- Operating temperature.

layer has been formed, one at the surface of grains and the other at the boundary between ZnO/SnO₂. Due to Au doping, the depletion layer expands, and the oxygen ion adsorption increases. Subsequently, decreased electron concentration increases the surface resistance and improves the gas-sensing response. The ZnO concentration of 1:0.5 records a maximum response of 100 for ethanol gas. Wang *et al.* [174] reported 3 wt% Ag-ZnO NFs to have a good response approximately 5 times faster than pristine ZnO NF. They observed the sensing response of Ag-ZnO NF increases with the rise in temperature and the maximum response is recorded at 320 °C. The increased response of Ag-ZnO fiber is attributed to the smaller size (3–5 nm) effect of Ag nanoparticles as compared to the thickness of the depletion layer of pure ZnO (approximately 15 nm) besides the increased surface area. However, the surface area change is small and the increase in response is due to the small size of Ag nanoparticles. The maximum response for ethanol is much larger than for acetone, formaldehyde, and toluene. From the above literature, increased surface area, adsorption of oxygen molecules, depletion layer thickness, and multiple depletion layers are the changes due to the doping of various elements with ZnO, which leads to better sensing.

5.3 Ammonia gas sensors

n-ZnO/p-NiO heterostructure NF was spun on a glass substrate by Lokesh *et al.* [175] and maximum response to ammonia was found with increased response upon an increase in ammonia concentration. This response is attributed to the ammonia's ability to donate electrons to the conduction band of ZnO, which increases the conductivity of the NF. Alali *et al.* [176] synthesized pure ZnO, CoNiO₂-ZnO, and CoNiO₂-ZnO hollow NFs on an aluminum substrate by electrospinning. The surface area difference is observed in all three types of NFs. The BJH result confirms the presence of 5–25 nm diameter of

pores in the pure ZnO, 5–10 nm in CoNiO₂-ZnO solid NF, and CoNiO₂-ZnO hollow NF. The increase of ammonia sensing response is reported at a temperature from 80 to 420 °C, and the highest response of the gas sensor recorded is 240 for CoNiO₂-ZnO hollow NF at 220 °C. It is four times greater than pure ZnO, and selectivity investigation reveals CoNiO₂-ZnO hollow NF has the highest response for ammonia gas compared to other test gases. The porous structure and large surface area help to increase the gas sensing performance. Andre *et al.* [177] fabricated pure ZnO and poly (styrene sulfonate) decorated ZnO NF. They observed necklace-shaped nanoparticles, which confirms the presence of a high surface area with the porous structure on the NFs which favors the suitable sensing property of the NFs. The SO₃²⁻ ions in the PSS create a strong interaction between the SO₃²⁻ ions and the NH₃ gas. This results in absorbing more NH₃ when it meets the surface of ZnO NFs, changing the charge state of PSS and improving the sensing performance. Doping of PSS with the ZnO improves the charge mobility between the ZnO NF and the ammonia gas which increases the sensor response. The 50% PSS-ZnO NF shows the best response, good selectivity, stability, and recovery time for NH₃ compared to NO₂ and CO gases.

5.4 Hydrogen gas sensors

Katoch *et al.* [178] observed many 30 nm-sized uniformly distributed nanograins on the ZnO and SnO₂ NFs. When the H₂ gas is exposed to the sensor, the metallic transformation of ZnO can be observed. Due to this transformation, the depletion layer is diminished and electron flow takes place from the metallic Zn to the surface of ZnO, resulting in a lowering of the resistance and enhancing the sensor response. The response of the H₂ gas increases as the temperature rises with a maximum obtained at 350 °C. Response and sensitivity of

SnO_2 NF are lower as compared to that of ZnO NF. During the exposure of H_2 gas, a resistance change in the p-n junction of SnO_2/ZnO results in the release of the chemisorbed oxygen molecules into the conduction band of ZnO. Both SnO_2 and ZnO NFs show good H_2 gas response with sensitivity compared to other gases like toluene, CO, and benzene. Hyoung *et al.*^[179] observed a rough surface with fine nanograins on NiO-doped ZnO NF. The sensing response increases with doping up to 0.05 wt% and decreases with further increased concentration of SnO_2 . The sensor's highest response is recorded at a temperature of 200 °C for H_2 gas. A decrease in resistance is recorded as the temperature increases. This change is due to the transfer of a large number of electrons to the conduction band from the ZnO valence band. When the temperature rises, response and recovery time decrease due to faster gas diffusion. The selectivity results show a high response to H_2 gas compared to benzene and carbon monoxide. Lee *et al.*^[112] reveal the presence of nanograins on the surface of 0.05 Co_3O_4 doped ZnO calcinated NF. They observe a good crystallinity structure of the NF. When the surfaces of ZnO and Co_3O_4 come in contact, the electrons tend to flow from ZnO to Co_3O_4 forming a potential barrier (electron and hole). When H_2 gas is introduced, it combines with the Co_3O_4 to form water molecules leaving behind Co. Since the work function of ZnO (5.2 eV) is lower than Co_3O_4 (6.3 eV), the current on the surface flows through the Co layer to ZnO nanograins. This results in lowering the resistance and enhancing the sensing properties. As the temperature increases to 300 °C, the response increases then decreases as the temperature moves higher which is due to the release of electrons from the conduction band. The responses and sensitivity are highest for H_2 gas compared to carbon monoxide and nitrogen dioxide. The sensor's response and recovery time decrease with higher sensing temperature and Co_3O_4 doping.

5.5 Hydrogen sulfide gas sensors

Akash *et al.*^[180] observed small nanograins on ZnO-CuO NF and found the nanograin's size increases with an increase in calcination duration. The NF with smaller nanograins has stronger H_2S sensing capabilities than those with larger nanograins. The band bending at the p-n junction results in forming a potential barrier for electrical conduction. When the gas is introduced, the copper oxide reacts with the H_2S forming copper sulfide, resulting in slumping of the depletion region and expediting the electron flow from CuS to ZnO, resulting in the decrease of resistance. When the gas is stopped and the sensor is exposed to dry air, the copper sulfide is converted into a copper oxide which recovers the p-n junction interface resulting in high resistance. A decrease in the sensor response is observed with the increase of nanograins present in the fiber. The maximum response for H_2S is obtained for 0.07 wt% CuO doped at 150 °C. Zhaorui *et al.*^[181] synthesized ZnO- SnO_2 with a 20.1 nm average crystalline size of ZnO and 19.8 nm for SnO_2 . When the sensor is in contact with air, the

depletion layer forms around the ZnO surface, capturing electrons from the conduction band and increasing sensor resistance. Likewise, when the H_2S gas is exposed, it reacts with the adsorbed oxygen and releases the captured electrons back to the conduction band, reducing the boundary barrier, which decreases resistance. Sensor response increases as temperature rises, with maximum response recorded at 250 °C. The sensor responses are measured at a concentration of 0.5–100 ppm, and the results show a linear relation at optimum temperature. The dynamic response of SnO_2 -doped ZnO NF to H_2S gas is observed from 1 to 50 ppm, and the results show a higher response as the concentration increases. The response and recovery time of the sensor are 18 s and 32 s, which indicates that the sensor reacts quickly and returns to its initial value with exposure to the air again, resulting in good stability and reproducibility. The repeatability of the sensor for H_2S gas is observed for 30 days at 250 °C. A minimal change in the response indicates the high stability of the sensor. Wang *et al.*^[174] observed the 3 wt% Ag-doped ZnO sensor has a greater response than the pure ZnO. This is because of the spillover effect where more oxygen molecules are passed from Ag nanoparticles to the ZnO surface. Also, with the electron sensitization effect, Ag adsorbs oxygen ions on the surface and generates oxygen species (O_2^- , O^- and O^{2-}). These two effects create a thicker depletion layer in Ag-ZnO compared to pristine ZnO NF which is beneficial for the adsorption of ethanol.^[182] The 3 wt% Ag-doped ZnO sensor records response and recovery times of 529 s and 690 s. The dynamic response to different concentrations of H_2S gas (1–10 ppm) is observed, and the results show the response values increase upon increasing the gas concentration. The sensor exhibits good repeatability for different periods with consistent response values and response/recovery time. When compared to ethanol, acetone, formaldehyde, and toluene at 120 °C, the sensors have a high response value to H_2S indicating good selectivity.

5. Conclusions

The level of sensing of a particular gas by the doped ZnO NF depends on surface area and resultant resistance change when it is in contact with that gas molecule. This aspect depends on the electronic, type of dopant used, and structure-property changes due to the particular dopant concentration. Hence, the proper selection of dopant and its concentration to fabricate ZnO-based NF for a particular gas sensing is essential. In addition to these electrospinning parameters, calcination temperature and duration also influences the response. The morphology of the NF surface is influenced by calcination temperature and the process parameter of electrospinning. The literature shows that the detection of gas molecules is found effective due to porosity, rough surface, and enormous, small nanograins, which increase the effective surface area of the NF. Also, the effective surface area per unit volume of NF is high for the small size of NF with a hollow structure. Designing a highly stable and efficient sensor for industrial-scale applications requires having the metric of long-term stability

without dropping the initial response. Several strategies are adopted for improved sensitivity of ZnO NFs, but only a few of them are suitable for long-term endurance. Additionally, large surface area and porosity are vital for producing sensors at a large-scale usage, which could provide high sensitivity and response with portable size. Many sensors described in this paper are fabricated and tested at a lab scale, while mass production sensors are challenging due to cost-effectiveness. Interdisciplinary collaborative research efforts are needed for the wide applications of resistive sensors.

6. Future scope of work

From the literature review, it is found that a large number of ZnO NFs-based gas sensors have been investigated. Out of which, it is noticed that fabricating a sensor device with a lower operating temperature is the demand in the current scenario in addition to high sensing response, good selectivity, and long-term stability. Furthermore, several attempts to improve the gas-sensing response of ZnO NFs have been made. This includes the functionalization of noble metals, doping of transition metals, the inclusion of carbonaceous nanomaterials, nanocomposites, and high-energy irradiation on ZnO NFs. It is also found that these methods are efficiently helping in reducing the operating temperature due to the introduction of extra adsorbed oxygen ions and significant change in the depletion layer in the presence of target gases. However, at RT, ZnO NFs-based gas sensors have poor reversibility and a long response/recovery time, making them ineffective for the early detection of target gases. The long-term stability of ZnO-based gas sensors is another challenge attributed to changing environmental conditions like different temperatures and different humidity levels. Hence, focused research is essential in ZnO NF-based research to develop gas sensors to overcome the above-said limitations.

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Conflict of interest

There are no conflicts to declare.

Supporting information

Not Applicable.

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Abbreviations

NFs	Nanofibers
ES	Electrospinning
ZnO	Zinc Oxide
Al ₂ O ₃	Aluminum Oxide
NP's	Nanoparticles
NiO	Nickel Oxide
TiO ₂	Titanium Dioxide
CA	Cellulose Acetate
Co ₃ O ₄	Cobalt Oxide
ppm	Parts per million
MB	Methylene blue
PVA	Polyvinyl alcohol
CO	Carbon monoxide
CO ₂	Carbon dioxide
C ₃ H ₆ O	Acetone
C ₂ H ₅ OH	Ethanol
H ₂	Hydrogen
UV	Ultraviolet
HF's	Hollow Fibers
H ₂ S	Hydrogen sulfide
NH ₃	Ammonia
Ni	Nickel
Co	Cobalt
SnO ₂	Tin Oxide
V ₂ O ₅	Vanadium Pentoxide
ZnAc ₂	Zinc Acetate
nm	Nanometer
kV	Kilo volt
eV	Electron volt
Wt.	Weight
cP	Centipoise
PVDF	Polyvinylidene fluoride
mL	Milliliter
RH	Relative humidity
SAW	Surface acoustic wave
mm	Millimeter

WO ₃	Tungsten trioxide
SPV	Surface photo voltage
RT	Room temperature
NO ₂	Nitrogen dioxide
O ₂	Oxygen
C ₆ H ₆	Benzene
CH ₄	Methane
NO	Nitric Oxide
Pd	Palladium
Zn	Zinc
Au	Gold
Pt	Platinum
RDB	Rhodamine B
s	Seconds
Ce	Cerium
La	Lanthanum
Er	Erbium
CoNiO ₂	Cobalt Nickel oxide
PSS	Poly styrene sulfonate
CuS	Copper Sulphide
d	Needle tip - Collector distance
v	Voltage
t _c	Calcination temperature
t	Duration
d _f	Diameter of fiber
r	Response
t _o	Operating temperature
Cu	Copper
Mn	Manganese
CNF	Cellulose NF
rGO	Reduced graphene oxide
Fe	Iron
TiCl ₄	Titanium tetrachloride
FESEM	Field emission scanning electron microscopy
XRD	X-ray diffraction
XPS	X-ray photoelectron spectroscopy

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