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Publisher: Elsevier

ISSN: 2773-0131 E-ISSN: 2773-0123

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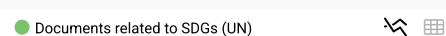
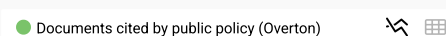
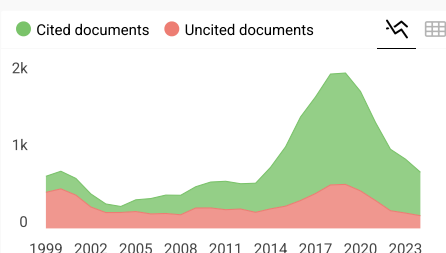
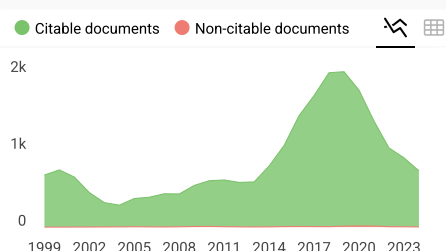
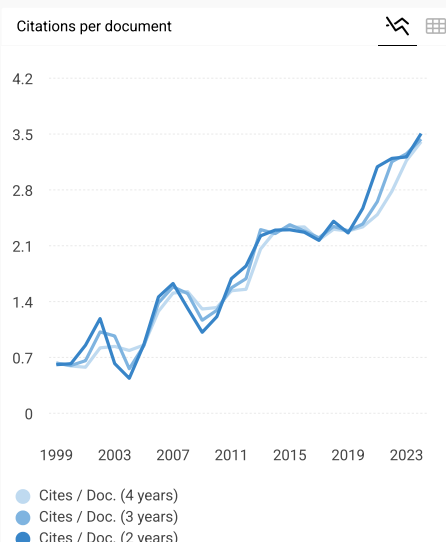
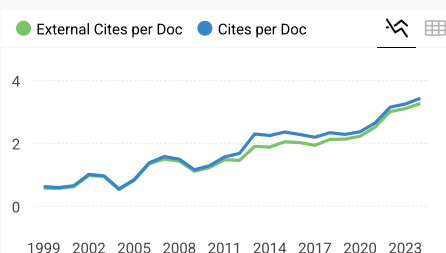
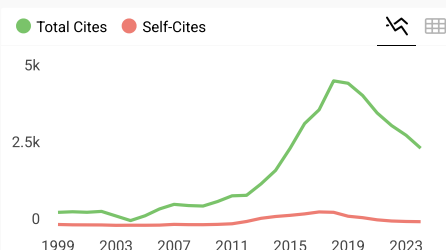
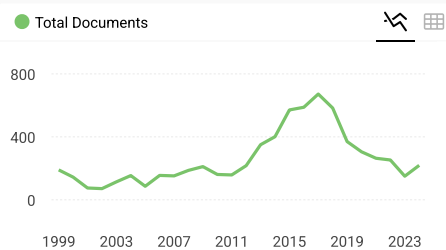
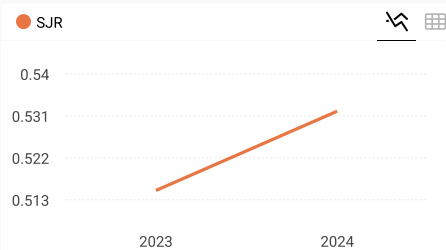
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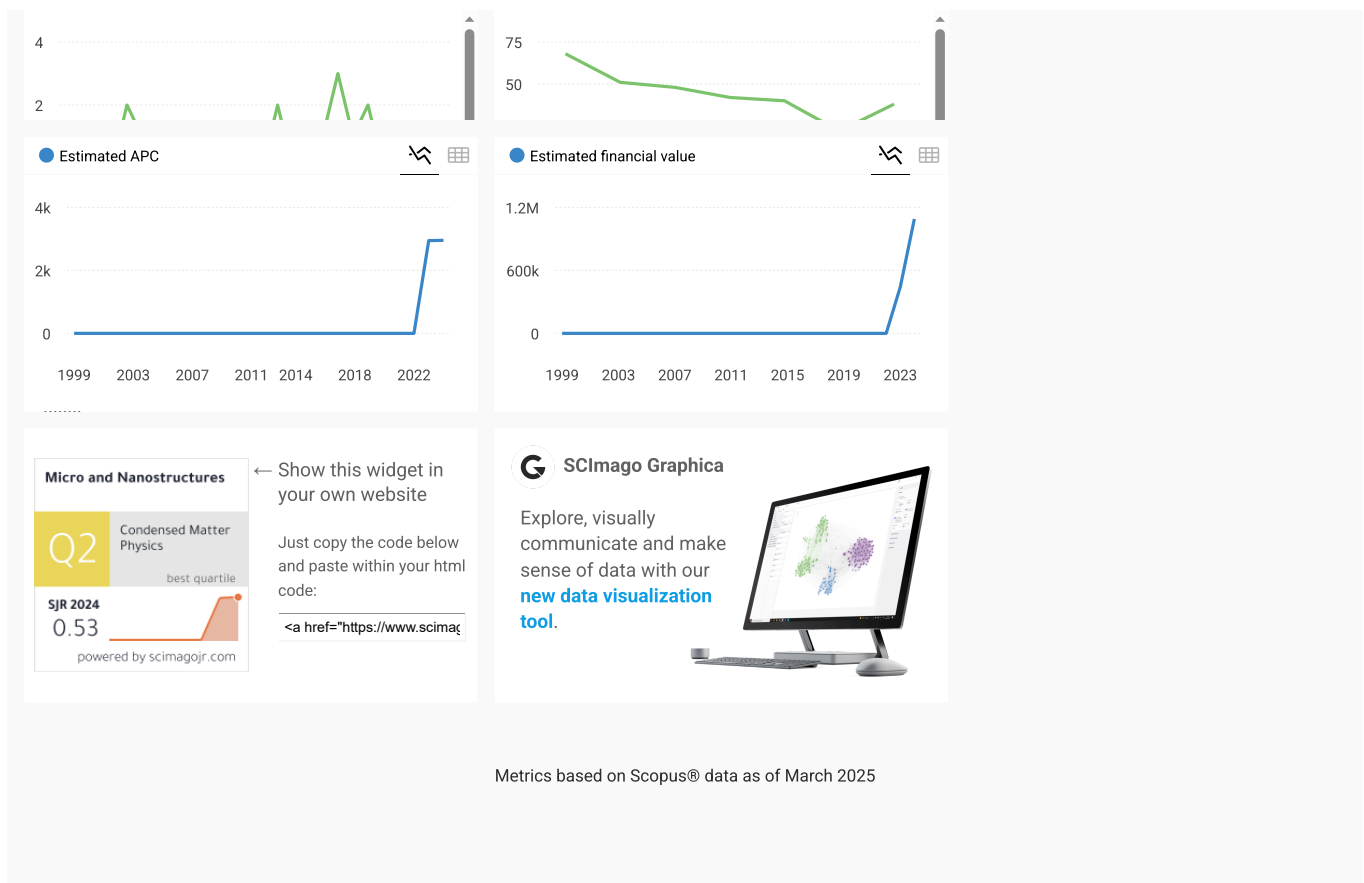
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Metal/Metal Oxide Nanoparticles Decorated Carbon Nanomaterials-Based Gas Sensors: A Review

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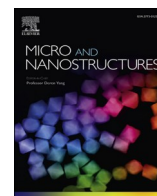
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Metal/metal oxide nanoparticles decorated carbon nanomaterials-based gas sensors: A review

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ARTICLE INFO

Keywords:

Gas sensor
Carbon nanomaterials
Metal/metal oxide nanoparticles
Gas/VOCs detection
Sensing materials

ABSTRACT

The advancement of high-performance gas sensors plays a crucial role in environmental protection, industrial safety, and public health. Carbon nanomaterials, including carbon nanotubes (CNTs), graphene, and carbon nanofibers (CNFs), have emerged as promising platforms due to their exceptional surface area, electrical conductivity, and chemical stability. However, their pristine forms often exhibit limitations in sensitivity, selectivity, and response dynamics. Recent advances have demonstrated that decoration of carbon nanomaterials with metal or metal oxide nanoparticles significantly enhances gas sensing performance by introducing catalytic activity, increasing active sites for gas adsorption, and promoting charge transfer. This review systematically explores the structural properties, synthesis methods, gas-sensing mechanisms, and application-specific advantages of these hybrid nanocomposites. Various synthesis techniques, including sol-gel, chemical vapor deposition (CVD), hydrothermal, and microwave-assisted processes, are discussed with emphasis on morphology control and functional optimization. Key findings reveal that heterojunction formation (e.g., p-n, Schottky interfaces) plays a pivotal role in tuning the sensor response. Comprehensive case studies illustrate the detection of hazardous gases such as NH₃, NO₂, H₂S, CO, and VOCs at room or low temperatures with high selectivity and sensitivity. The combination of metal/metal oxide nanoparticles and carbon nanostructures offers promising prospects for creating flexible, wearable, and portable gas sensors tailored for real-time and low-power applications.

1. Introduction

The rapid growth of the global population, which is currently increasing by approximately 83 million people each year, has led to urbanization and industrialization, resulting in the depletion of natural resources [1]. This rapid development has significantly affected environmental conditions, particularly by elevating air pollution levels. According to the World Health Organization (WHO), exposure to both outdoor and indoor air pollution causes an estimated seven million premature deaths each year, primarily due to respiratory and cardiovascular diseases [2]. Thus, real-time detection and monitoring of toxic and hazardous gases have become

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critically important for environmental protection, industrial safety, and public health. Gas sensors play a crucial role in detecting low concentrations of hazardous gases. An ideal sensor should combine high sensitivity and selectivity with fast response and recovery times, robust long-term stability, low power consumption, cost-effectiveness, and compatibility with flexible or wearable electronic systems [3–5]. Achieving these demanding criteria requires the development of advanced sensing materials with superior physico-chemical properties.

Carbon nanomaterials, including carbon nanofibers (CNFs), carbon nanotubes (CNTs), and graphene, have emerged as promising candidates for next-generation gas sensors owing to their high specific surface area, excellent electrical conductivity, superior mechanical strength, and outstanding chemical stability [5–8]. Their nanoscale dimensions provide abundant active sites for gas molecule adsorption while their excellent electronic properties facilitate the efficient conversion of chemical interactions into detectable electrical signals [7,9]. However, pristine carbon nanomaterials often have limitations, including poor selectivity towards specific gases, moderate sensitivity at room temperature, and slow response/recovery behavior [10,11]. To address these shortcomings, extensive research has focused on decorating carbon nanomaterials with metal/metal oxide nanoparticles. The decoration of carbon nanomaterials with metal nanoparticles such as gold (Au), platinum (Pt), and silver (Ag) as well as metal oxides such as tin oxide (SnO_2), tungsten trioxide (WO_3), and zinc oxide (ZnO) introduces catalytic sites, enhances gas adsorption, and promotes charge transfer processes leading to significantly improved sensing performance [12–18]. These hybrid sensors demonstrate potential for detecting various toxic gases such as NH_3 , NO_2 , H_2S , CO, and VOCs at room temperature and with high selectivity. Moreover, fabrication techniques such as hydrothermal synthesis, sol-gel processing, chemical vapor deposition (CVD), and microwave-assisted synthesis have facilitated the development of these nanocomposites with controlled morphology and desirable properties.

This review offers a comprehensive overview of recent progress in gas sensors utilizing metal/metal oxide nanoparticles-decorated carbon nanomaterials. It covers the fundamental properties of carbon nanomaterials, synthesis techniques, sensing mechanisms, sensor performance, and practical applications. The review offers valuable insights into the design and optimization of high-performance, flexible, and wearable gas-sensing technologies, addressing contemporary needs for environmental and health

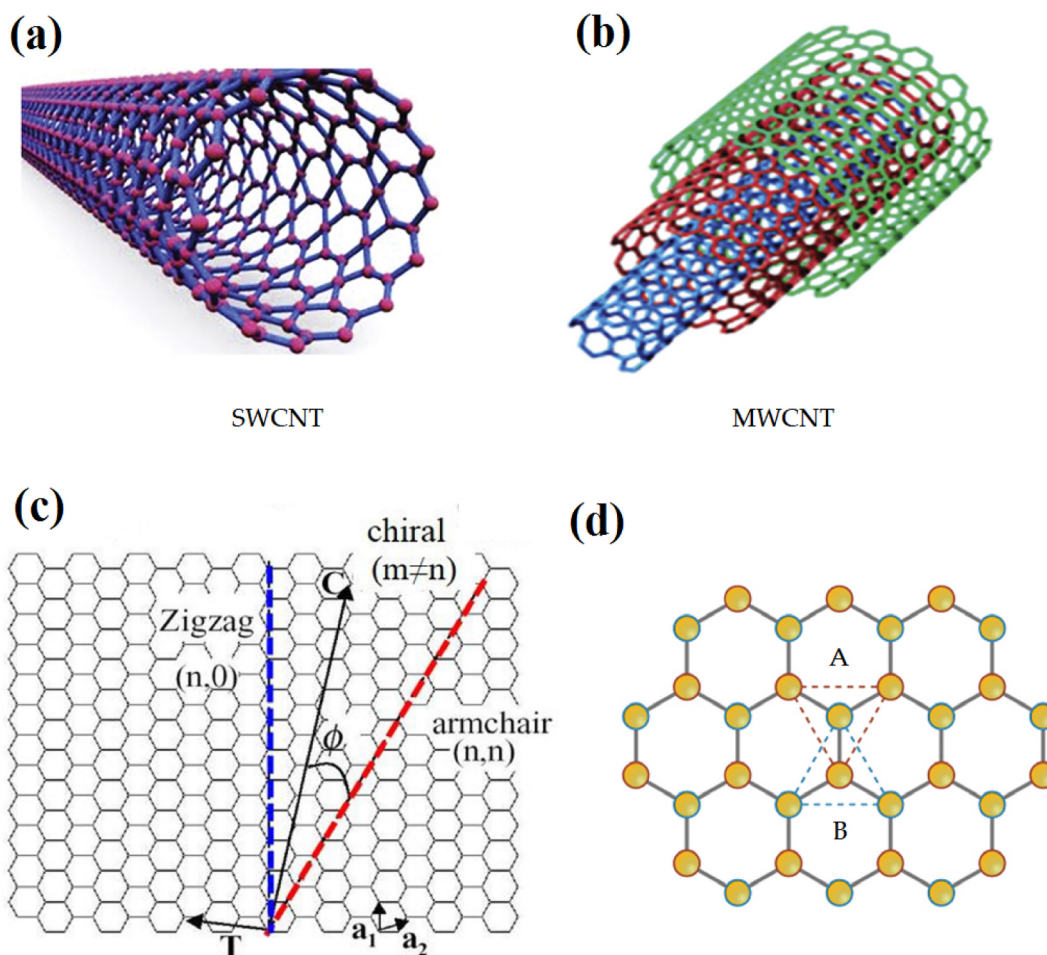


Fig. 1. Representation of (a) SWCNT and (b) MWCNT (Reproduced with permission from [35]). (c) Rolling directions of a graphene sheet along lattice vectors to form armchair, zigzag, and chiral SWCNTs [25]. (d) Graphene structure (Reproduced with permission from [56]).

monitoring.

2. Carbon nanomaterials and their properties

Carbon nanomaterials are a promising class of materials for applications in gas sensing. Their unique properties play an important role in enhancing the performance of gas sensors due to their nanoscale dimension and specific arrangements of carbon atoms. This section presents the key properties of some carbon-based nanomaterials often used for gas sensors, including CNTs, CNFs, graphene, and its derivatives.

2.1. Carbon nanotubes (CNTs)

CNTs are one-dimensional structures formed by rolling graphite sheets into cylindrical shapes, typically with diameters less than 100 nm and lengths extending up to several micrometers [3,19–21]. CNTs are generally classified into two types based on their wall structure: (i) single-walled CNTs (SWCNTs) and (ii) multi-walled CNTs (MWCNTs). The SWCNTs are formed from a single rolled graphene sheet, while MWCNTs comprise several concentric SWCNT layers as illustrated in Fig. 1a and b. In MWCNTs, the individual graphene cylinders are held together by a delocalized π -electron cloud resulting from the sp^2 hybridization of carbon atoms [22,23]. CNTs can have three structural types: armchair, zigzag, and chiral [22,24,25]. These types are characterized by a chiral vector denoted by a pair of indices (n, m) , which specify the direction in which the graphene sheet is rolled and determine the closure of the cylindrical ends, enabling differentiation among SWCNT structures [24]. When $m = n$, the SWCNTs are classified as armchair. When $m = 0$, they are referred to as zigzag. Most SWCNTs are chiral ($m \neq n$). Fig. 1c demonstrates how a graphene sheet can be rolled to form armchair, zigzag, and chiral SWCNTs. The translation vector T is aligned with the nanotube axis, while a_1 and a_2 represent the lattice vectors of the 2D-graphene sheet.

CNTs display outstanding properties, including mechanical strength, unique electrical conductivity, and various chemical properties [21,26–29]. Their extraordinary mechanical strength is attributed to the robust sp^2 hybridization of carbon-carbon bonds [30,31]. The elastic modulus of CNTs is approximately 1 TPa, comparable to that of diamond (1.2 TPa) and nearly five times greater than steel [32–35]. Additionally, CNTs have a low density of about 1.3 g/cm^3 [24,36].

The electrical properties of CNTs are excellent because of the one-dimensional quantum effect [25]. CNTs can electrically act as either a semiconductor or a metal depending on their diameter and chirality [37,38]. For semiconducting CNTs, the band gap decreases as the diameter increases [39]. These outstanding characteristics make CNTs acted as promising materials for various applications, including sensors [40–43], solar cells [44–46], medicine [47–49], and environmental solutions [50–52]. In gas sensor applications, the gas sensing mechanism of CNTs is primarily related to the adsorption and desorption of gas molecules [3,53]. The curved surfaces and high area/volume ratios of CNTs enhance the adsorption process by increasing the contact interface between gas molecules and the CNT surfaces [53]. This enhancement results in a high sensitivity of CNTs towards the adsorbed gas molecules. The

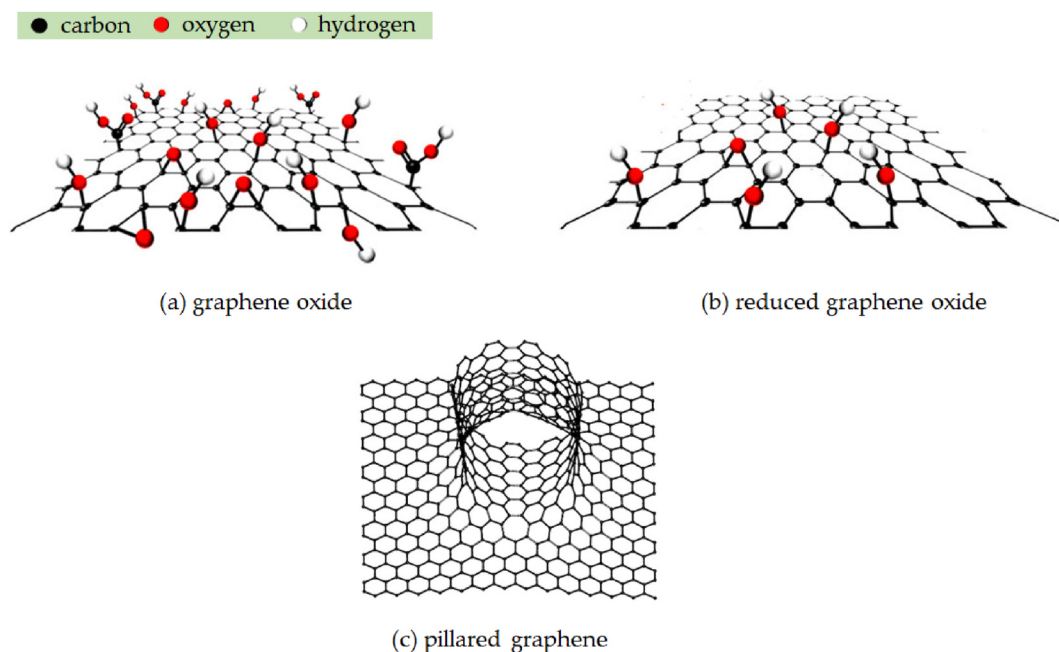


Fig. 2. Structures of some graphene derivatives: (a) graphene oxide, (b) reduced graphene oxide (Reproduced with permission from [72]) and (c) pillared graphene [73].

sensitivity of CNTs can be determined by measuring changes in electrical conductivity. Two mechanisms explain these changes: first, the conductivity of CNTs increases when charge is transferred from CNTs to gas molecules (hole donation). Alternatively, gas molecules donate electrons, which lowers the hole density on the CNT surface and results in decreased conductivity [3,54]. These changes in conductivity can be detected, making CNTs effective for gas/VOCs detection applications.

2.2. Graphene and its derivatives

Since the discovery of graphene in 2004 [55], it has garnered significant attention due to its remarkable properties. Graphene is structured as a 2D sheet of sp^2 -hybridized C atoms arranged in a honeycomb lattice, as shown in Fig. 1d. This structure consists of two interpenetrating triangular sublattices (red and blue triangles) and contains two carbon atoms in its unit cell [56]. The bond length between carbon atoms is 1.42 Å [56,57]. Graphene exhibits no band gap because of the overlap between the valence and conduction bands [58]. It shows a high electron mobility (above $15,000 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$) at room temperature and carrier concentrations of 10^{13} cm^{-2} [55], behaving as a semimetal or semiconductor. These characteristics provide graphene with unique electrical properties, making it suitable for applications in electronics, including integrated circuits, sensors, and transparent electrodes. In addition to its electrical properties, graphene has exceptional mechanical and thermal properties. Its thermal conductivity ranges from 3000 to 5000 $\text{Wm}^{-1}\text{K}^{-1}$ [58], while its mechanical strength is indicated by a Young's modulus of 1 TPa [59]. Optically, intrinsic graphene exhibits high opaqueness, with a low white light absorption of 2.3 % and reflectance of less than 0.1 % [60]. In addition, several graphene-based derivatives have been developed, such as graphene oxide (GO), reduced graphene oxide (rGO), pillared graphene, and graphene scrolls. These derivatives are classified based on layer number, dimensions, and carbon-oxygen ratios. Fig. 2 presents some structures of graphene derivatives. The unique properties of graphene and its derivatives make them suitable for a variety of applications across numerous fields, including the environment [61,62], medicine [63,64], energy [65,66], and intelligent materials [67, 68].

Among the derivatives of graphene, GO has attracted significant attention due to its abundance of oxygen-containing functional groups including hydroxyl, carboxyl, and epoxy groups. These functional groups enhance the material's hydrophilicity and dispersibility, facilitating better dispersion in solvents and making it more versatile for various applications [69]. The presence of these functional groups not only enhances the material's interaction with gas molecules but also increases its surface area, which is crucial for gas-sensing applications. The GO's ability to adsorb gas molecules through weak van der Waals forces or stronger chemical bonds due to these functional groups makes it particularly effective for detecting low concentrations of various gas molecules. Moreover, GO can be chemically or thermally reduced to form rGO, which restores conductivity while retaining some functional groups, further enhancing sensing performance. Importantly, the type and amount of oxygen functionalities strongly modulate sensitivity and selectivity. For example, hydroxyl and carboxyl groups commonly improve the adsorption of polar acidic or basic gases (e.g., NO_2 , NH_3), while epoxy groups and defect sites can act as stronger chemisorption centers for certain volatile organic compounds (VOCs) [70,71].

The important characteristics of graphene-based gas sensors include a large surface area-to-volume ratio, rapid response times, and favorable thermal and electronic properties [74]. These features enable graphene and its derivatives to detect gas molecules at concentrations as low as parts per billion (ppb) [75]. By introducing functional groups, the performance of graphene-based gas sensors in terms of sensitivity and selectivity can be significantly improved [76,77].

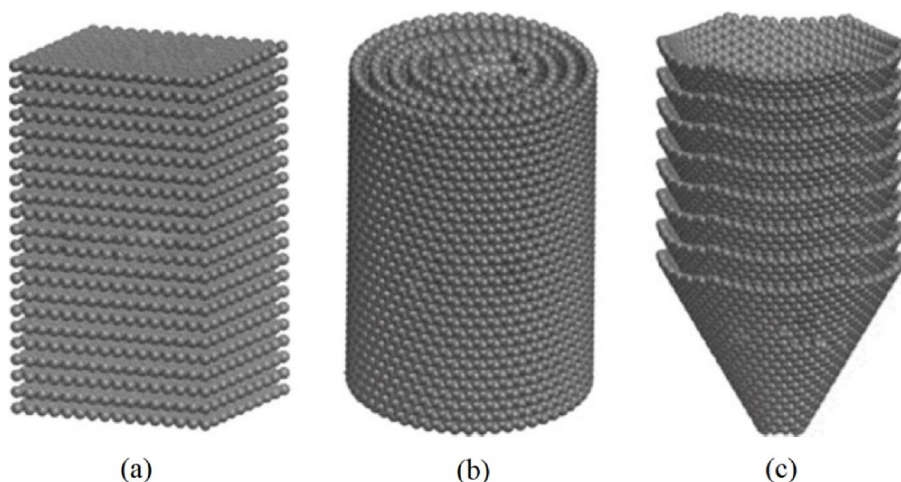


Fig. 3. A schematic diagram of three types of CNFs: (a) platelet, (b) tubular, and (c) fishbone. Reproduced with permission from [78].

2.3. Carbon nanofibers (CNFs)

Carbon nanofibers (CNFs) are one-dimensional nanomaterials, and their structures are more complicated than carbon nanotubes [78]. Their structures consist of layers of graphitic planes stacked in the longitudinal direction of the fibers and vary in the range of 5–100 μm in length and 20–60 nm in diameter [5]. There are three main types of CNFs (Fig. 3) including platelet-type, tubular-type, and fishbone-type [78]. CNFs exhibit high electrical conductivity, excellent thermal conductivity, and remarkable mechanical and chemical properties [78–80]. CNFs are useful for applications in which high electrical conductivity is required, such as electrode materials for batteries [81], supercapacitors [82], etc. Furthermore, their outstanding conductivity and chemical properties, combined with a high surface area, which provides large active sites, make them particularly attractive for gas sensing applications [83,84].

3. Surface functionalization of carbon nanomaterials for gas sensing

Surface functionalization is a vital technique for modifying carbon-based nanomaterials to enhance their performance and optimize their structure. By employing physical, chemical, or plasma treatments, functionalization alters the surface chemistry, introduces active sites, and improves compatibility with metal or metal oxide nanoparticles [85,86]. This process enhances gas adsorption, modulates charge transfer, and allows for adjustable sensitivity and selectivity. Typically, carbon nanomaterials, such as CNTs, graphene, and CNFs, are subjected to treatments like acid oxidation [87], plasma irradiation [86], or heteroatom doping [88]. These methods facilitate the incorporation of various functional groups (including $-\text{COOH}$, $-\text{OH}$, $-\text{NH}_2$, etc.) onto their surfaces [89]. These functional groups enhance gas adsorption by increasing active binding sites, improving surface polarity, and creating defect sites that serve as charge-transfer channels between adsorbed gas molecules and the conductive carbon framework.

For instance, surface functionalization of MWCNTs with a combination of oxygen plasma treatment, gold nanoparticle decoration, and 16-mercaptohexadecanoic acid (MHDA) self-assembled monolayers could tailor the chemical affinity and selectivity of CNTs toward specific VOCs. This multilayer modification enhanced sensitivity to non-aromatic gases (alcohols and acetone), reduced interference from humidity and NO_2 , and improved response and recovery behavior, demonstrating the critical role of surface chemistry in optimizing gas sensor performance [90]. Similarly, nitrogen-doped rGO showed higher sensitivity and excellent reproducibility in NO gas sensing at room temperature compared to pristine rGO. It is attributed to the enhanced catalytic activity due to the increased number of active sites, including nitrogen atoms at the edges and in heterocyclic rings of graphene [91]. In the case of CNFs, functionalizing them with gold nanoparticles led to improved performance for ethanol gas sensing at room temperature. This enhancement included a higher response, a faster response time, and greater stability compared to the pristine one. These benefits are due to the combination of the high surface area of the CNFs and the catalytic activity of gold nanoparticles [92]. These findings show that surface functionalization controls molecular interactions and greatly impacts the performance regulation and structural optimization of carbon nanomaterials for gas sensing.

4. Metal/metal oxide nanoparticles for gas sensing

4.1. Metal nanoparticles

Metal nanoparticles exhibit unique properties such as a high surface-to-volume ratio due to their small size, a significant percentage of atoms or molecules on the surface, catalytic activity, and tunable electronic properties [93–96]. These characteristics of metal nanoparticles contribute to a wide range of applications [93,97,98]. In gas-sensing applications, metal nanoparticles provide numerous active sites for gas molecule interactions owing to their high surface area, thereby enhancing the sensitivity of gas sensors. Furthermore, their small size and the abundance of surface atoms enhance the reactivity of these nanoparticles, facilitating the adsorption and reaction with specific gas molecules. Metal nanoparticles, such as silver (Ag), copper (Cu), gold (Au), platinum (Pt), and palladium (Pd), have attracted significant interest for their ability to enhance the sensing performance of carbon nanomaterials [99–104]. For example, Au is well-known for its high stability and is effective in detecting gases like carbon dioxide [104]. Pd is particularly useful for hydrogen sensing because of its ability to adsorb and dissociate hydrogen molecules [95]. Pt serves as a highly effective catalyst and is frequently used in hydrogen sensing applications [99]. Additionally, Ag has excellent electrical conductivity and is useful for detecting carbon dioxide and ammonia [103].

4.2. Metal oxide nanoparticles

Metal oxide nanoparticles are widely recognized for their effectiveness in gas-sensing applications attributed to their unique physicochemical properties and sensing mechanisms. Their large surface area, abundant active sites, and high surface activity enable metal oxide nanoparticle-based gas sensors to exhibit high sensitivity and rapid response/recovery [6,105]. Metal oxide materials can be categorized as n-type or p-type depending on the nature of their dominant charge carriers—free electrons in n-type and holes in p-type materials. These charge carriers are fundamental to the gas-sensing mechanism, as interactions between gas molecules and the material surface induce changes in resistance or conductivity. Examples of n-type metal oxide nanoparticles include zinc oxide (ZnO) and tin oxide (SnO_2), which are commonly used in gas sensors to detect gases like CO, NH_3 , and H_2 [6,106,107]. On the other hand, p-type metal oxide nanoparticles include copper oxide (CuO) and nickel oxide (NiO) which can be used for sensing applications involving gases such as carbon dioxide (CO_2), hydrogen, and nitrogen dioxide (NO_2) [6,106–109]. Nowadays, metal oxide nanoparticles like ZnO, SnO_2 , TiO_2 , CuO, NiO, and WO_3 are extensively researched for their application in decorating carbon nanomaterials

to enhance the performance of gas sensors [110–114].

5. Advantages of metal/metal oxide nanoparticles decorated carbon nanomaterials for gas sensing

Gas sensors utilizing pure carbon nanomaterials can face limitations such as limited selectivity, reduced sensitivity, and slow response and recovery times [4]. However, incorporation of metal or metal oxide nanoparticles onto their surfaces can significantly improve their sensing efficiency. These nanoparticles exhibit a high surface area to volume ratio and physicochemical properties [97] leading to enhanced sensing performance of carbon nanomaterials. The important roles of metal/metal oxide nanoparticles in improving the gas sensing performance of carbon nanomaterials are as follows.

5.1. Enhanced sensitivity

One of the major advantages of decorating carbon nanomaterials with metal or metal oxide nanoparticles is the significant enhancement in gas sensitivity. In gas sensing, sensitivity refers to a sensor's capability to detect changes in gas concentration, often down to ppm or even ppb levels. When carbon-based nanomaterials such as CNTs or graphene are functionalized with metal or metal oxide nanoparticles, the sensitivity is further improved due to several contributing factors.

5.1.1. Increased surface area

Metal nanoparticles increase the surface area for gas adsorption. Nanoparticles are generally small and can be distributed evenly over the carbon nanomaterial, exposing more active sites where gas molecules can adsorb. This increases the interaction between gas molecules and the sensor surface, leading to improved sensitivity [6,104].

5.1.2. Electron transfer and charge carrier modulation

The interaction between gas molecules and metal nanoparticles integrated into carbon nanomaterials significantly influences the electronic properties of these composites, leading to notable changes in their electrical conductivity. Metal nanoparticles like silver (Ag), gold (Au), or platinum (Pt) [99,100,103] can serve as electron acceptors or donors, facilitating charge transfer between the gas molecules and the carbon matrix. This modulation of charge carriers significantly changes the electrical conductivity of the material, which is easily detectable as a change in sensor signals.

5.1.3. Chemical affinity of metal nanoparticles

Many metal and metal oxide nanoparticles exhibit a strong affinity for specific gases. This chemical affinity can lead to the selective adsorption of particular gas molecules, thereby enhancing the sensor's sensitivity to those gases. For instance, palladium (Pd) nanoparticles have a high affinity for hydrogen (H_2), which significantly increases the sensitivity of Pd-doped carbon nanomaterials to hydrogen gas [94].

5.2. Catalytic activity

Metal and metal oxide nanoparticles function as catalysts, facilitating the interaction between gas molecules and carbon nanomaterials. This catalytic activity is crucial in enhancing the sensor responses and sensitivity. Specifically, metal oxide nanoparticles, such as tin oxide (SnO_2), zinc oxide (ZnO), and titanium dioxide (TiO_2), are well-known for their ability to catalyze the oxidation or reduction of gases, making them highly effective in gas-sensing applications [6,110–112,115].

5.3. Improved selectivity

Decorating carbon nanomaterials with metal or metal oxide nanoparticles enhances the chemical specificity of gas sensors, improving their selectivity toward particular gases [116]. While pure carbon nanomaterials such as CNTs or graphene are generally sensitive to a wide range of gases, the incorporation of specific metal or metal oxide nanoparticles can target the detection of certain gases, making the sensors more selective. By fine-tuning the type and concentration of metal or metal oxide nanoparticles, it is possible to customize the sensor responses to a specific target gas, reducing cross-sensitivity and improving overall detection accuracy. This chemical specificity is key to developing highly selective gas sensors for a variety of applications.

5.4. Faster response and recovery times

Incorporating metal/metal oxide nanoparticles significantly accelerates the gas adsorption and desorption processes, leading to shorter response and recovery times for gas sensors. This enhanced dynamic behavior is particularly crucial for real-time gas detection and monitoring applications, where fast detection and recovery are essential for ensuring sensor performance and reliability. Metal nanoparticles such as palladium (Pd), platinum (Pt), and silver (Ag) can improve the kinetics of gas adsorption and desorption by providing additional active sites on the sensor surface, which facilitate faster interaction with gas molecules. These nanoparticles also promote rapid electron transfer between the adsorbed gas molecules and the sensor material, enabling faster signal generation and recovery [117–120].

6. Synthesis of metal/metal oxide nanoparticles decorated carbon nanomaterials for gas sensors

The synthesis of metal/metal oxide nanoparticles decorated carbon nanomaterials involves multiple strategies, including hydrothermal methods, sol-gel methods, microwave-assisted synthesis, chemical vapor deposition, and others. Each of these methods has distinct advantages and limitations, which can significantly impact the final characteristics of the nanocomposites. In this section, we present the synthesis techniques for carbon nanomaterials decorated with metal/metal oxide nanoparticles.

6.1. Hydrothermal methods

Hydrothermal synthesis is a widely used technique for fabricating carbon nanomaterials that are decorated with metal or metal oxide nanoparticles, which are particularly effective in gas sensing applications. This method involves the reaction of precursors, typically metal salts and carbon sources, within a sealed vessel, such as a Teflon-lined autoclave. The process is conducted at elevated temperatures, usually between 100 and 250 °C, and under high pressure. The unique hydrothermal environment promotes the nucleation and growth of well-crystallized nanostructures with controlled morphologies, particle sizes, and enhanced surface areas. In hydrothermal processes, various parameters, such as temperature, pressure, pH, reaction time, and precursor concentration, can be finely tuned to tailor the physical and chemical properties of the resulting nanocomposites. This precise control enables the formation of different nanostructures, including nanorods, nanosheets, nanospheres, and hierarchical architectures. These structures are crucial for gas sensing due to their large surface-to-volume ratios and abundant active sites [121,122]. For example, Ding et al. [123] synthesized zinc oxide–reduced graphene oxide (ZnO–rGO) nanocomposites via a hydrothermal method and utilized them as an electrochemical sensor for detecting hydrazine. Three different morphologies of ZnO on the rGO surface were obtained by varying the mass ratio of Zn^{2+} to rGO. The fabricated electrochemical hydrazine sensor based on ZnO–rGO nanocomposites synthesized by the hydrothermal method exhibited high sensitivity and excellent selectivity toward hydrazine. Lin et al. [124] synthesized SnO_2 –graphene (SnO_2/G) composites via a simple one-pot hydrothermal method at 120 °C for 8 h. Graphene oxide served as a template, while SnCl_2 acted as the precursor. The synthesized composite displayed a three-dimensional nanostructure featuring flower-like microspheres of SnO_2 nanoflakes interspersed among layers of graphene, which were decorated with tiny SnO_2 nanoparticles, as illustrated in Fig. 4a and b. The synthesized SnO_2/G composites were used to fabricate gas sensors for ammonia sensing, as shown in Fig. 4c.

6.2. Chemical vapor deposition (CVD)

Chemical vapor deposition (CVD) is a commonly employed method for producing carbon-based nanomaterials like CNTs and

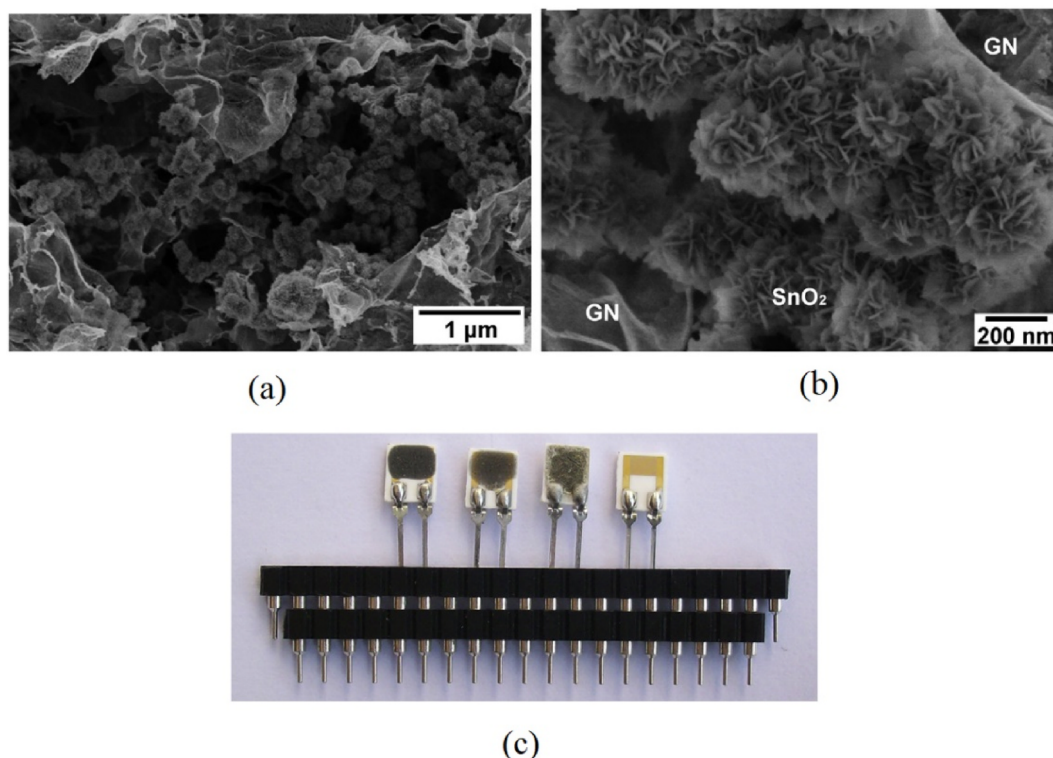


Fig. 4. SEM images of (a), (b) SnO_2/G composites and (c) photo of SnO_2/G based gas sensors. Reproduced with permission from [125].

graphene, which can subsequently be decorated with metal/metal oxide nanoparticles to improve their gas-sensing performance. In this method, gaseous precursors containing carbon are introduced into a heated reaction chamber, where they decompose at high temperatures, typically between 600 °C and 1000 °C. This process results in the deposition of carbon atoms on a substrate, forming nanostructured materials. To enhance the gas-sensing capabilities of these carbon nanomaterials, they can be decorated with metal or metal oxide nanoparticles. This decoration can be achieved by introducing metal or metal oxide precursors during the CVD process, allowing for the simultaneous growth of nanoparticles on the carbon structures [125,126]. The CVD method offers the advantage of precise control over various parameters, including pressure, temperature, gas flow rates, and the ratio of carbon to metallic precursors. This control enables the tailoring of the morphology and size of both the carbon nanomaterials and their metal or metal oxide coatings, which is crucial for optimizing their performance in gas sensing applications.

6.3. Microwave-assisted synthesis

In this method, microwaves serve as the energy source for the reaction. This technique provides a rapid, energy-efficient, and highly controllable means of producing carbon nanomaterials decorated with metal or metal oxide nanoparticles. The process typically involves mixing carbon precursors with metal or metal oxide sources in a microwave-transparent vessel. Upon exposure to microwave radiation, these materials undergo enhanced chemical reactions that promote the nucleation and growth of nanoparticles on the carbon matrix. This results in composite materials with improved surface area, conductivity, and catalytic properties. Additionally, this technique significantly reduces reaction times compared to conventional methods, and microwave irradiation often enables the simultaneous reduction of metal precursors without requiring external reducing agents [127–129]. For example, Kim et al. [130] fabricated NO₂ gas sensors using graphene-SnO₂ nanocomposites with a commercial microwave oven. Briefly, the mixture of SnO₂ nanopowders and graphene was initially dispersed in ethanol for 1 h. After dispersion, the mixture was dried on filter paper. The prepared dried powder was then placed into an alumina crucible and treated using a microwave process at a power of 1 kW for 5 min, with the radiation time varied from one to 8 min. Fig. 5a illustrates the synthesis process of graphene-SnO₂ nanocomposites using

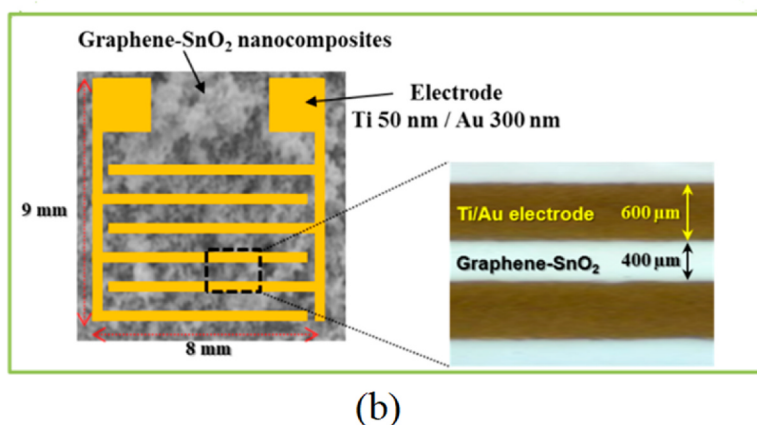
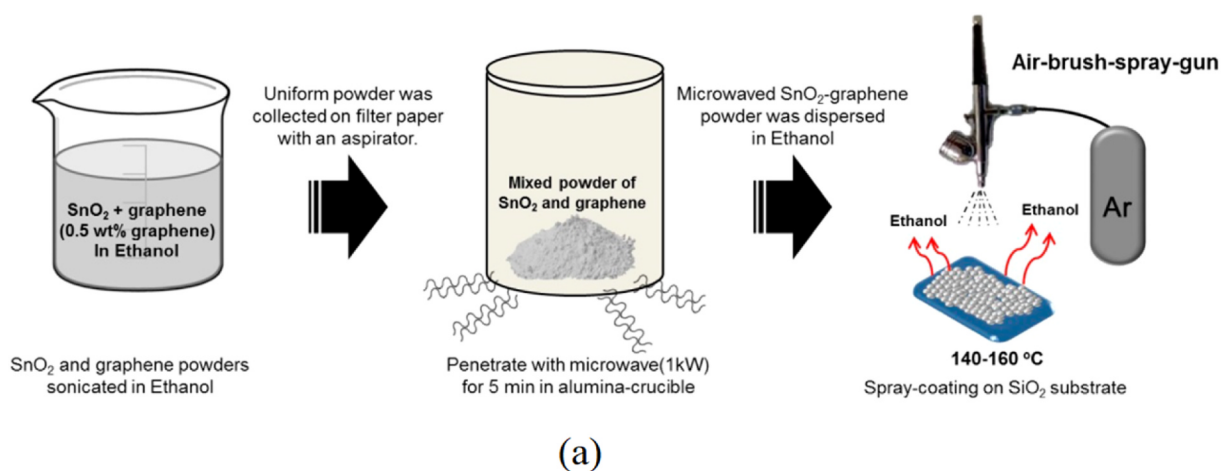


Fig. 5. Illustration for (a) synthesis of graphene-SnO₂ nanocomposites using microwave-assisted synthesis and (b) fabricated gas sensors. Reprinted with permission from [131].

microwave-assisted synthesis. Subsequently, the synthesized nanocomposites were used to fabricate gas sensors, as shown in Fig. 5b.

6.4. Sol-gel methods

The sol-gel method is a versatile and cost-effective bottom-up chemical synthesis route widely used for producing carbon nanomaterials decorated with metal oxides or metal nanoparticles, providing precise control over composition, morphology, and microstructure. It relies on the hydrolysis and polycondensation of metal alkoxide or metal salt precursors, resulting in the formation of a sol (colloidal suspension) that gradually transitions into a gel (three-dimensional network) under controlled conditions [131]. This gel matrix facilitates the uniform nucleation of nanoparticles onto carbon nanomaterials. Subsequently, aging, drying, and thermal treatment improve the crystallinity of nanoparticles and interfacial bonding. The incorporation of carbon nanomaterials, such as graphene, carbon nanotubes, into the sol phase results in hybrid nanocomposites with synergistic properties: the carbon matrix provides high electrical conductivity, large specific surface area, and mechanical stability, while metal oxides such as SnO_2 , ZnO , and TiO_2 supply abundant active sites for gas adsorption and catalytic reactions [105,132]. Key advantages of the sol-gel method include low processing temperatures, uniform dopant distribution, tunable porosity, and the ability to create homogeneous nanocomposites with tailored porosity and surface chemistry [133]. Additionally, its compatibility with soft and flexible substrates broadens the potential for developing flexible or wearable sensor platforms.

7. Mechanisms of gas sensing

The gas-sensing mechanism typically involves the processes of adsorption and desorption, which occur between gas molecules and the surfaces of sensing materials. Two primary mechanisms of gas adsorption, chemisorption (chemical adsorption) and physisorption (physical adsorption), play significant roles in gas sensing [134,135]. These mechanisms differ in the strength of the interactions between gas molecules and the surfaces of sensing materials. Chemisorption involves the formation of strong chemical bonds, such as covalent and ionic bonds between the gas molecules and the sensing materials [134]. In contrast, physisorption is characterized by weaker interactions such as van der Waals forces and hydrogen bonding [134]. Chemisorption is ideal for gas sensors that need high sensitivity and selectivity for detecting specific gases, although it may result in slower response and recovery times. On the other hand, physisorption is well-suited for fast and reversible gas detection, especially in situations where higher gas concentrations are anticipated. Still, it might be less sensitive compared to chemisorption-based methods.

In the case of carbon nanomaterials, they exhibit remarkable electronic properties that make them highly effective for gas-sensing applications. Understanding the behavior of charge carriers (electrons and holes) in these materials is essential for grasping their sensing mechanisms. When gas molecules interact with the surface of carbon nanomaterials, they affect the concentration and mobility of charge carriers, resulting in detectable changes in electrical properties such as conductivity or resistance [75,136]. Carbon nanomaterials, including carbon nanotubes and graphene, exhibit semiconducting characteristics that can be tailored to achieve either n-type (electron-conducting) or p-type (hole-conducting) behavior. Generally, oxidizing gases (electron acceptors), such as NO_2 and NO , withdraw electrons from the carbon nanomaterials, whereas reducing gases (electron donors) like NH_3 donate electrons to them [134,137].

In p-type carbon nanomaterials, conductivity increases when exposed to oxidizing gases due to charge transfer from the sensing material to the gas molecules, resulting in a higher concentration of holes [137]. In contrast, reducing gases lower the conductivity of the sensing material because electrons are transferred from the gas molecules to the sensing material, resulting in a lower concentration of holes [138,139]. In n-type carbon nanomaterials, the changes in conductivity occur opposite, with oxidizing gases decreasing conductivity and reducing gases increasing it. Additionally, the large surface area of carbon nanomaterials provides numerous active sites for enhancing the interaction between gas molecules and the materials and enables integration with other sensing materials [8].

Metal and metal oxide nanoparticles have emerged as essential components in gas-sensing technology due to their high surface

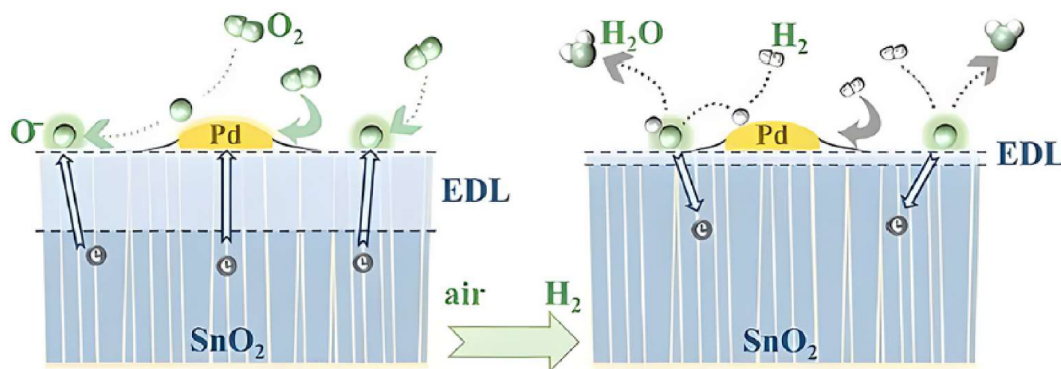


Fig. 6. The catalytic properties of Pd nanoparticles on the SnO_2 surface [105].

area, adjustable surface chemistry, and capability to detect a wide range of gases with high sensitivity [6,93,105,120]. The small size and high surface-to-volume ratio of these nanoparticles, which increase the number of active sites for gas molecule adsorption, enhance their interaction with gas molecules [140]. Moreover, these nanoparticles' catalytic properties facilitate chemical reactions with gas molecules. This property also enhances the gas-sensing mechanism by promoting oxidation or reduction reactions [113,141,142]. The gas-sensing mechanism in metal and metal oxide nanoparticles is primarily based on the interaction between the gas molecules and the nanoparticle surface, which induces measurable changes in the material's electrical properties. For metal nanoparticle-based gas sensors, when gas molecules interact with the surface of metal nanoparticles, they either donate or accept electrons, depending on the type of gases (oxidizing or reducing) [143,144], leading to charge transfer and altering the electronic properties of the nanoparticles. For instance, NO_2 is a strong oxidizing gas that accepts electrons from the surface of metal nanoparticles, creating a depletion region and increasing sensor resistance [134]. Due to their catalytic properties, metal nanoparticles act as catalysts that enhance oxygen adsorption onto material surfaces and facilitate the dissociation of gas molecules [95,120]. As shown in Fig. 8, SnO_2 doped with Pd nanoparticles is fabricated for the H_2 gas sensor. When the sensing material is exposed to air, the Pd nanoparticles help facilitate the dissociation of the O_2 molecules. Then, the dissociated oxygen atoms subsequently diffuse from the Pd nanoparticles to the SnO_2 surface and accept free electrons from the conduction band of SnO_2 , forming adsorbed oxygen anions and creating an electron depletion layer (EDL) [95]. For H_2 detection, as shown on the right side of Fig. 6, Pd nanoparticles dissociate H_2 molecules into hydrogen atoms [145]. Interaction between the diffused hydrogen atoms and adsorbed oxygen anions results in the narrowing of the electrical double layer (EDL).

In metal oxide-based gas sensors, the semiconducting properties of metal oxides allow their electrical conductivity of the materials to change when the composition of the surrounding atmosphere changes [105]. The principles of the gas-sensing mechanism are based on the changes in resistances of the sensing materials from the adsorption of oxygen ions [3,146]. When metal oxides are exposed to air between 200 and 500 °C (working temperature for metal oxide) [3], oxygen molecules in the air are adsorbed on the surface of metal oxides and trap electrons in the conduction band of the oxides, forming adsorbed oxygen species (O_2^- , O^- , O^{2-}). When electrons in the conduction band of the oxides become trapped by the oxygen species, an electron depletion layer forms on the surface of n-type metal oxides, while a hole accumulation layer forms on the surface of p-type metal oxides [120]. Thus, the resistances of metal oxides vary, increasing in n-type metal oxides and decreasing in p-type metal oxides [120]. When gas molecules interact with the surface of metal oxides, they engage with the pre-adsorbed oxygen species, leading to changes in the material's electrical resistance. In the case of reducing gases like NH_3 , this interaction releases electrons back into the conduction band of the metal oxide, resulting in an increased electron concentration and a thinner electron depletion layer, which lowers the resistance of n-type metal oxides. Conversely, exposure to oxidizing gases extracts electrons from the conduction band, enlarging the depletion layer and thereby increasing the resistance of n-type metal oxides [147]. To investigate the gas-sensing mechanism of p-type metal oxides, Fig. 7 illustrates a nickel oxide (NiO)-based gas sensor designed for the detection of hydrogen gas. The hole accumulation layer develops when the sensor is exposed to air. When exposed to hydrogen gas (a reducing gas), the adsorbed oxygen species interact with the gas via redox reaction [6], releasing electrons into the conduction band of NiO. The released electrons recombine with holes in the hole accumulation layer of NiO (electron-hole recombination) [108]. As a result, hole concentration in the hole accumulation layer decreases, leading to an increase in the resistances of NiO. Conversely, an oxidizing gas introduces additional holes to the hole accumulation layer by extracting more electrons, which leads to a reduction in the resistance of p-type metal oxides.

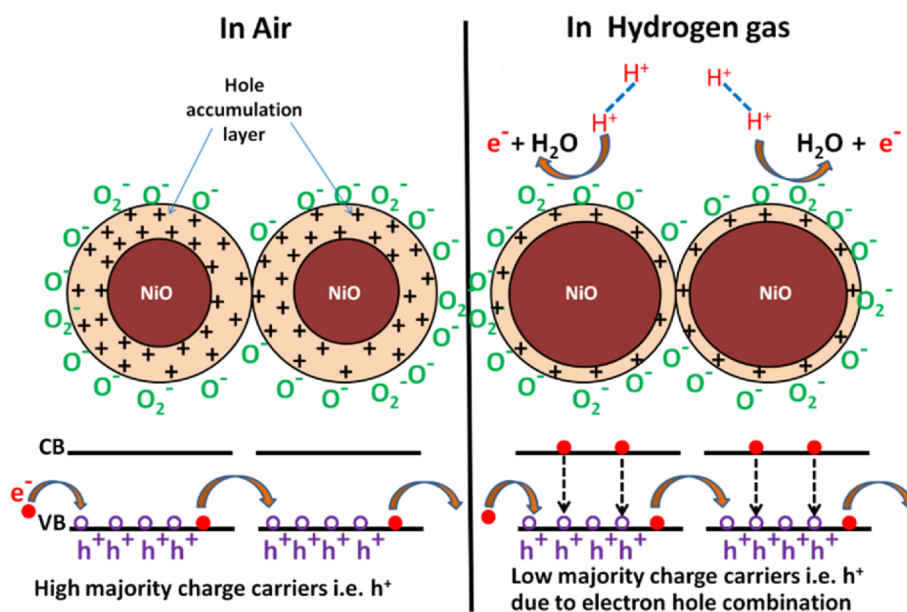


Fig. 7. Gas-sensing mechanism of a NiO-based gas sensor for hydrogen gas detection. Reproduced with permission from [109].

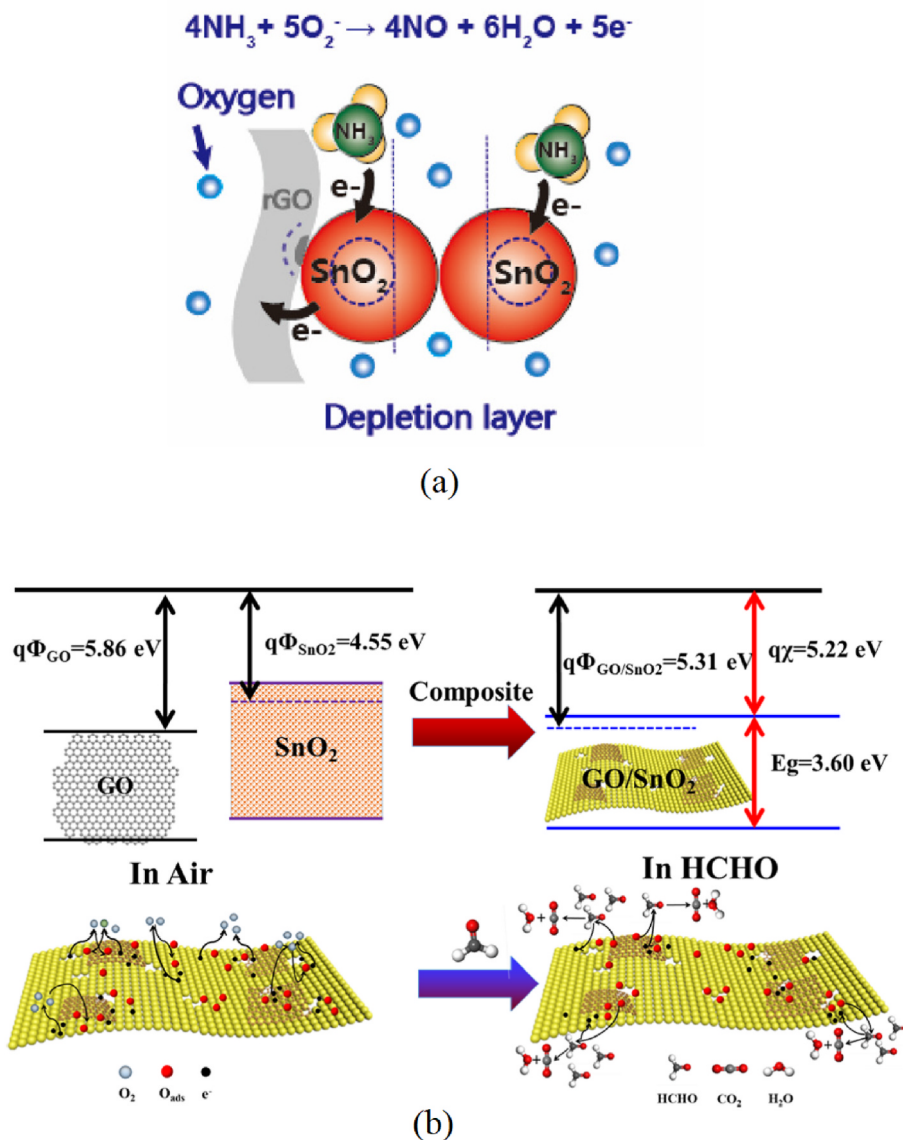


Fig. 8. Schematic diagrams of gas-sensing mechanism for (a) rGO/SnO₂ in the presence of NH₃ (Reproduced with permission from Ref. [149]) and (b) GO/SnO₂ in the presence of HCHO (Reprinted with permission from Ref. [152]).

The incorporation of metal or metal oxide nanoparticles into carbon nanomaterials has emerged as a powerful strategy for enhancing high-performance gas sensors [103,104,113]. When carbon nanomaterials are combined with metal/metal oxide nanoparticles, they form a heterojunction at the interface, such as p-p, n-n, p-n, and Schottky heterojunctions [6,120,148]. These hybrid nanocomposites combine the properties of both materials for effective gas sensing. The gas-sensing mechanisms primarily involve changes in electrical properties, adsorption, catalytic activity, synergistic effect, and charge transfer processes [149]. Metal/metal oxide nanoparticles play a crucial role in facilitating adsorption and interaction with gas molecules due to their catalytic properties. Carbon nanomaterials enhance electron mobility and provide an interconnected network for charge transfer. Among various heterojunction types, the p-n heterojunction can modulate the width of the electron depletion layer, which enhances the gas sensing capabilities of nanocomposite-based sensors. When the p-n heterojunction forms at the interface of the hybrid nanocomposites due to the difference in their Fermi levels, electrons diffuse from the n-type materials to the p-type materials while holes move in the opposite direction until their Fermi levels reach the same energy level [150]. This movement of both electrons and holes results in the formation of a depletion layer on the n-side and an accumulation layer on the p-side. The redistribution of charges creates an internal electric field and a potential barrier at the interface which significantly affects charge carrier transport across the junction [151]. The gas sensing mechanism in these hybrid nanocomposites relies on the modulation of the potential barrier at the interface and the charge transfer processes that occur during the adsorption and desorption of gas molecules. In ambient air, oxygen molecules are adsorbed onto the

surface of the n-type materials, capturing free electrons and forming ionic oxygen species. This process enhances the depletion layer in n-type materials, thereby increasing their resistance. Conversely, for the p-type carbon nanomaterials, oxygen adsorption leads to hole accumulation resulting in a decrease in resistance. At the p–n heterojunction, the built-in potential restricts the flow of charge carriers and the sensor reaches a quasi-steady-state baseline resistance determined by the combined effects of surface adsorption and junction characteristics. The resistance of the p–n junction can be altered by detecting gas molecules. In the case of reducing gases, these gas molecules donate electrons to the conduction band of the n-type materials when they interact with the adsorbed oxygen species, decreasing both the resistance and the depletion width of the junction [150,151]. In contrast, oxidizing gases extract electrons, which increases the built-in potential and widens the depletion layer. This results in higher junction resistance, hindering carrier transport.

Fig. 8a displays the sensing mechanism of rGO/SnO₂-based gas sensors for detecting NH₃ at room temperature. Two depletion layers are formed: one at the surface of the SnO₂ nanoparticles and another at the p-type rGO/n-type SnO₂ interface (p–n heterojunction). When exposed to NH₃, the oxygen species adsorbed on the surface of the nanoparticles interact with the NH₃ gas molecules, which act as electron donors. This interaction releases electrons into the conduction band of SnO₂. The electrons in the conduction band of SnO₂ are then transferred to the rGO nanosheets, significantly enhancing the adsorption of NH₃ [149]. Furthermore, the transfer of electrons from NH₃ to SnO₂ reduces the depletion layer between the SnO₂ nanoparticles, resulting in a decrease in resistance. This is attributed to the fact that SnO₂ nanoparticles act as a catalyst for the rGO nanosheets, facilitating the adsorption of NH₃ at room temperature.

In the case of using GO/SnO₂ sensing materials for HCHO detection [152], as shown in Fig. 8b, the p–n heterojunctions form when GO is decorated with SnO₂, creating a new energy level structure. When exposed to air, adsorbed oxygen species accumulate on the surface of GO/SnO₂, leading to layers of electron depletion and an increase in the resistance of the sensitive materials. In the presence of HCHO, the adsorbed oxygen species interact with the HCHO molecules, and the interaction releases electrons back to the conduction band, decreasing the material resistance. The porous and ultrathin structure of the GO/SnO₂ hybrid materials increases surface area and active sites, promoting interactions with HCHO gas molecules and achieving extremely high responses. In addition, the ultrathin nanosheet structure of GO provides shortened transport paths, which significantly enhance the response of the sensing hybrid materials. Moreover, the plentiful pores in the SnO₂ structure facilitate gas diffusion, contributing to improved response and recovery performance. Also, GO serves as a spacer that reduces the agglomeration of SnO₂ nanoparticles and provides additional adsorption sites for HCHO gas molecules, ultimately resulting in enhanced gas responses.

8. Applications of metal/metal oxide nanoparticles decorated carbon nanomaterials for gas-sensing

8.1. Carbon nanotubes decorated with metal/metal oxide nanoparticles

8.1.1. CNTs decorated with metal nanoparticles

CNT-based gas sensors have been studied for the past two decades due to their unique properties for gas sensing applications, and they are easy to synthesize, compact, and inexpensive. Furthermore, their electronic properties are highly sensitive to any change in a chemical environment. The sensitivity of CNTs to gas molecules depends on the number of CNT walls [7]. However, pristine CNTs often exhibit low sensitivity and selectivity. To enhance these characteristics, CNTs are decorated with metal or metal oxide nanoparticles, which improve the interaction between the gas molecules and sensing material. Various metal nanoparticles are used to decorate the surface or edges of CNTs. Noble metal nanoparticles, including Ag, Au, Pt, and Ti, are the most popular. Lin et al. [12] studied the use of Au nanoparticles to enhance the sensitivity of CNTs for gas detection. CNTs were grown on a Si substrate at 700 °C by thermal chemical vapor deposition (CVD), and C₂H₂ was used as a carbon source. Au nanoparticles were then coated onto the CNT networks via the sputtering technique [91] with a thickness of 5 nm. The Au-decorated CNT-based sensors were used to detect carbon dioxide, ethanol, and isopropyl alcohol. The sensor responses increased with increasing gas concentrations. Compared to pristine CNTs, those decorated with Au nanoparticles exhibit significantly higher responses to target gases. This indicates that the presence of Au on the CNT surface enhances the gas sensitivity of CNT-based sensors, likely due to catalytic effects.

In the case of Ag decoration, Fam et al. [13] investigated the enhancement of hydrogen sulphide (H₂S) gas detection using SWCNTs decorated with Ag nanoparticles at room temperature. The sensing material demonstrated strong sensitivity and excellent selectivity toward H₂S, with minimal cross-sensitivity to other gases like carbon monoxide and nitric oxide. This selectivity is attributed to the strong affinity between Ag and sulfur atoms, facilitating specific interactions with H₂S molecules.

Pd-decorated CNTs have been extensively investigated for the detection of toxic gases, such as NH₃, CO, and H₂S. Choi et al. [153] developed gas sensors by decorating CNTs with Pd nanoparticles to detect NH₃ and CO at room temperature. The sensors exhibited a significant increase in resistance upon exposure to NH₃, indicating electron donation to the CNTs. In contrast, exposure to CO led to a slight decrease in resistance in pure CNT sensors, whereas CNT-Pd sensors showed an increase, suggesting electron withdrawal due to the catalytic properties of Pd nanoparticles. The CNT-Pd sensors demonstrated fast response times (less than 16 s), linear sensitivity, and low detection limits, 7 ppm for NH₃ and 20 ppm for CO, along with good repeatability and minimal noise. These characteristics highlight the potential of CNT-Pd sensors as effective and simple tools for detecting toxic gases at room temperature. Choi et al. [154] reported the development of highly sensitive and selective NO₂ gas sensors based on SWCNTs decorated with Pt nanoparticles. The Pt nanoparticles were deposited onto the SWCNTs using a sputtering technique, followed by thermal annealing to optimize particle dispersion and interaction with the CNT surface. The fabricated sensors exhibited superior NO₂ sensing performance at room temperature compared to pristine SWCNT sensors. Specifically, the Pt decoration significantly increased sensitivity, reduced response/recovery times, and enhanced selectivity toward NO₂ over other interfering gases such as CO, NH₃, benzene, and toluene. The improved gas sensing performance resulted from the catalytic activity of Pt nanoparticles, which enhanced the adsorption and partial

dissociation of NO_2 molecules into more active chemical species, facilitating effective charge transfer between the gas molecules and the SWCNTs.

8.1.2. CNTs decorated with metal oxide nanoparticles

CNTs decorated with metal oxide nanoparticles have emerged as highly effective hybrid materials for gas-sensing applications. They combine the remarkable electrical conductivity and high surface area of CNTs with the chemical reactivity and selectivity of metal oxides [19,20]. This synergistic integration enhances gas adsorption, facilitates charge transfer, and often results in hetero-junction formation that significantly improves sensor response [7]. Humayun et al. [155] studied the fabrication of methane gas sensors through the functionalization of MWCNTs with ZnO nanoparticles using atomic layer deposition (ALD) techniques. The heterostructures of ZnO nanoparticles and MWCNTs were formed, with ZnO nanoparticles deposited onto the active sites of the CNT

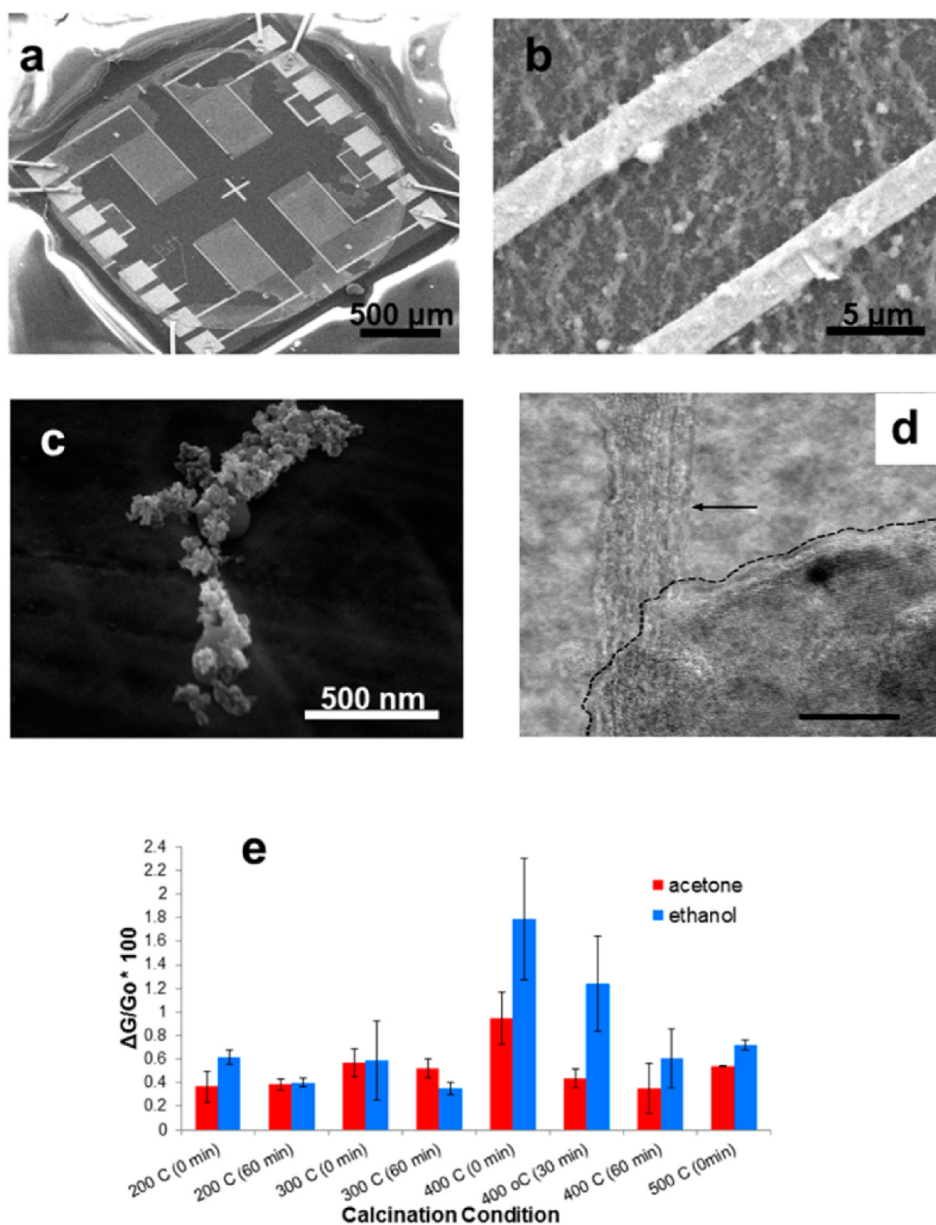


Fig. 9. SEM images of (a) a silicon substrate featuring four interdigitated gold electrodes, (b) two electrodes bridged by an $\text{In}_2\text{O}_3/\text{SWCNTs}$ network, and (c) the morphology of the $\text{In}_2\text{O}_3/\text{SWCNTs}$ composite. (d) HRTEM image of $\text{In}_2\text{O}_3/\text{SWCNTs}$ (the black arrow indicates bare SWCNTs and the dotted line indicates borders of In_2O_3 nanoparticles), and (e) sensing response of $\text{In}_2\text{O}_3/\text{SWCNTs}$ at various calcination conditions to 10 ppm ethanol and 9 ppm acetone. Reprinted with permission from [158]. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

surface. The sensor could detect methane concentration down to 2 ppm at room temperature. The authors concluded that functionalizing ZnO nanoparticles improves electron transport in the CNTs-ZnO junction, resulting in enhanced sensitivity for methane detection. Nahiriak et al. [156] studied the sensing properties of gas sensors fabricated from SnO₂-MWCNTs nanocomposites towards H₂. The SnO₂ nanoparticles were synthesized by CVD and sol-gel methods. Then, the nanocomposites used as a sensing layer were synthesized by ultrasonic mixing of SnO₂ nanoparticles (prepared via CVD or sol-gel methods) with oxidized MWCNTs. The sensors were exposed to H₂ concentrations ranging from 1000 to 10000 ppm. The findings indicated that the sensor response to H₂ was influenced by the percentage of CNTs present in the nanocomposites. Ellis et al. [157] presented the development of a hybrid material combining indium oxide (In₂O₃) nanoparticles with oxidized SWCNTs for efficient ethanol vapor detection at room temperature. Utilizing a sol-gel synthesis method, In₂O₃ nanoparticles were grown on the surface of oxidized SWCNTs, forming a core-shell nanostructure of In₂O₃/SWCNT composite with high conductivity and sensitivity. The researchers investigated the impact of calcination temperature on the material's sensing performance and found that intermediate calcination conditions (400 °C) yielded the best sensitivity to ethanol and acetone vapors as shown in Fig. 9. Fig. 9a–c shows SEM images of the developed sensor composed of four interdigitated gold electrodes with a layer of In₂O₃/SWCNT composite deposited by drop-cast method. Bare SWCNTs and borders of In₂O₃ nanoparticles are presented in Fig. 9d.

Several studies of carbon nanotubes decorated with metal or metal oxide nanoparticles for applications in gas sensing are summarized in Table 1.

8.2. Graphene and its derivatives decorated with metal or metal oxide nanoparticles

Graphene and its derivatives, including graphene oxide and reduced graphene oxide, provide an ultra-high surface area, exceptional electrical conductivity, mechanical strength, and chemical stability. These features are essential for effective gas sensor performance. However, pristine graphene typically shows limited selectivity and sensitivity toward specific gas molecules. Researchers have investigated enhancing graphene and its derivatives by decorating them with metal or metal oxide nanoparticles to overcome these limitations.

8.2.1. Graphene and its derivatives decorated with metal nanoparticles

Metal nanoparticles play a crucial role in enhancing the gas-sensing capabilities of graphene and its derivatives by introducing active sites that improve gas adsorption, charge transfer, and catalytic interactions. Moreover, some metal nanoparticles help dissociate gas molecules, resulting in better adsorption. Manna et al. [14] reported the fabrication of resistive sensors using Pt nanoparticles supported on rGO for detecting formaldehyde (HCHO) at room temperature. The results indicated that the presence of Pt nanoparticles enhanced the response of the Pt-rGO nanomaterial-based sensors by nearly 4.5 times compared to bare rGO sensors. The improved response results from the catalytic properties of Pt nanoparticles, which enhance the surface reactions with adsorbed HCHO. Similarly, Phan et al. [163] developed a hydrogen sensor with enhanced sensitivity and rapid response characteristics. The researchers synthesized Pt nanoparticles loaded onto three-dimensional (3D) graphene from graphene oxide and a Pt precursor using a polymer-assisted hydrothermal method. This Pt-decorated 3D graphene was then integrated onto a microheater to fabricate the hydrogen sensor as presented in Fig. 10. The fabricated sensor exhibited high sensitivity, fast response and recovery times, minimal hysteresis, and good linearity in detecting hydrogen gas. The enhanced performance is attributed to the catalytic properties of the Pt nanoparticles, which facilitate hydrogen adsorption and dissociation, as well as the porous 3D graphene structure that provides a large surface area and efficient electron transport pathways. This synergistic combination underscores the potential of Pt-decorated 3D graphene in developing high-performance hydrogen sensors for safety applications.

Ghanbari et al. [164] developed a methane gas sensor by decorating graphene with silver nanoparticles (AgNPs), achieving

Table 1

Applications of carbon nanotubes decorated with metal or metal oxide nanoparticles for gas-sensing.

Sensing materials	Target gases	Response/concentration	Operating temperature (°C)	Response/recovery times	Ref.
Au/CNT	CO ₂ , ethanol, isopropyl alcohol	1.2 % (CO ₂)/50 ppm, 1.2 % (ethanol)/50 ppm, 1.1 % (isopropyl)/50 ppm	RT	–	[12]
Au/CNT	NO ₂ , H ₂ S, NH ₃	~2.4 % (NO ₂)/0.5–10 ppm	200	–	[102]
Ag/SWCNT	H ₂ S	~2 ppm	RT	–	[13]
Pd/CNT	NH ₃ , CO	~7 ppm (NH ₃), 20 ppm (CO)	RT	8–16 s/-	[153]
Pt/SWCNT	NO ₂	63 %/2 ppm	100	–	[154]
ZnO/MWCNT	CH ₄	~2 ppm	RT	–	[155]
SnO ₂ /MWCNT	H ₂	~1000 ppm	140	–	[156]
In ₂ O ₃ /SWCNT	ethanol, acetone	~2 ppm (ethanol), 0.5 ppm (acetone)	RT	–	[157]
ZnO/CNT	CO ₂	3.6 %/30 sccm	RT	82.5 s/23 s	[158]
ZnO/MWCNT	CO	24 %/40 ppm	RT	8 s/-	[159]
ZnO/MWCNT	ethanol	~2.00/500 ppm	RT	18 s/29 s	[160]
SnO ₂ /CNT	NO ₂	~80/1 ppm	–	–	[161]
Pt, Ti, Ag, Ru, Cu/MWCNT	NO ₂ , H ₂ S, NH ₃ , CO	–/–	RT	–	[93]
Cu, Co/SWCNT	NH ₃	5.7 % (Cu), 5.9 % (Co)/2 ppm	RT	–	[162]

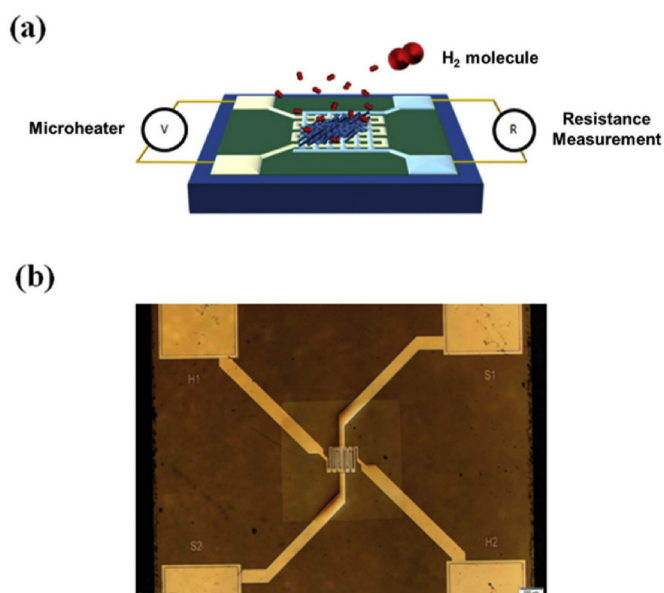


Fig. 10. (a) Concept of hydrogen sensor based on Pt-3D graphene and (b) an image of the fabricated microheater for H_2 sensor. Reproduced with permission from [165].

effective detection at low temperatures. The AgNPs, averaging 29.3 nm in size, were uniformly distributed on the graphene surface without aggregation. The sensor's performance was evaluated across various silver-to-graphene mass ratios (SGMRs) and methane concentrations under ambient conditions. An optimal SGMR of approximately 12 % was identified, enhancing the sensor's response to methane. For methane concentrations below 2000 ppm, the sensor showed a rapid and linear increase in response, even at room temperature. Ovsianyskyi et al. [165] reported a highly sensitive and selective H_2S gas sensor by decorating chemical vapor deposition-grown graphene with silver nanoparticles (AgNPs) and charged impurities. The fabrication involved immersing the graphene in an aqueous solution of $AgNO_3$ and $Fe(NO_3)_3$ for 4 min, during which Ag^+ ions were reduced to Ag^0 nanoparticles, while Fe^{3+} ions introduced charged impurities. These dopants generated active sites for H_2S adsorption and dissociation on the surface of graphene, leading to significant changes in resistivity upon gas exposure. The sensor demonstrated real-time H_2S detection at ambient temperature with an immediate response, a detection limit below 100 parts per billion (ppb), and consistent performance over multiple cycles. The sensing mechanism is based on charge transfer interactions between H_2S molecules and doped graphene which modify the charge carrier density and produce detectable changes in resistance.

Some studies have shown the development of gas sensors using graphene with metal co-doping or bimetallic functionalization. Kumar et al. [166] developed a hydrogen gas sensor utilizing a composite of palladium–platinum (Pd–Pt) nanoparticles dispersed on graphene layers. The composite was fabricated through chemical methods, resulting in isolated Pd–Pt alloy nanoparticles uniformly

Table 2

Applications of graphene and its derivatives decorated with metal or metal oxide nanoparticles for gas-sensing.

Sensing materials	Target gases	Response/concentration	Operating temperature ($^{\circ}C$)	Response/recovery times	Ref.
Pt/rGO	HCHO	7.5 %/25 ppm	RT	12 min/350 s	[14]
Pt/3D graphene	H_2	16 %/10000 ppm	200	9 s/10 s	[163]
Ag/graphene	CH_4	0–3.9 %/50–16000 ppm	25–125	—/—	[164]
Ag–Fe/graphene	H_2S	37 %/500 ppb	RT	350 s/-	[165]
Pd–Pt/graphene	H_2	5.1 %/2 % H_2	40	2 s/18 s	[166]
SnO_2 /graphene	H_2	6 %/100 ppm	50	1.1 s/1.1 s	[167]
In_2O_3 –ZnO/graphene	CH_4	–27.48 %/500 ppm	RT	48 s/169 s	[173]
CuO/rGO	CO_2	2.56 Hz/ppm/50 ppm	RT	41 s/20 s	[168]
SnO_2 /rGO	NO_2	65.5 %/5 ppm	RT	12 s/17 s	[169]
TiO_2 /rGO	NH_3	0.62/10 ppm	RT	55 s/-	[170]
V_2O_5 /rGO	NO_2	51 %/100 ppm	150	102.29 s/778.23 s	[174]
Ni–ZnO/rGO	H_2	63.8 %/100 ppm	150	28 s/-	[175]
ZnO, TiO_2 , SnO_2 /rGO	NO_2 , NO, SO_2	—/—	RT	—/—	[176]
TiO_2 /rGO	<i>n</i> -butanol	18/85 ppm	RT	352 s/506 s	[177]
Sn– TiO_2 @rGO/CNT	NH_3	85.9 %/250 ppm	RT	99 s/66 s	[178]
CuO/GO	CO_2	60 %/-	—	—/—	[179]
WO_3 /graphene	NO_2	133/5 ppm	250	25–200 s	[171]
ZnO/GONR	NO_2	~18/50 ppm	RT	31 s/27 s	[172]

distributed on graphene. The sensor demonstrated exceptional performance, achieving a rapid response time of less than 2 s and a recovery time of 18 s when exposed to 2 % hydrogen at 40 °C and 1 atm pressure. These rapid changes are due to hydrogen-induced modifications in the work function of the Pd–Pt alloy, as well as alterations in the distribution of defect states within the graphene band gap caused by gas adsorption.

From the studies mentioned above, metal nanoparticles significantly enhance the gas-sensing performance of graphene and its derivatives by introducing catalytically active sites that improve gas adsorption, charge transfer, and molecular dissociation processes. These studies highlight that metal nanoparticle decoration, whether monometallic or bimetallic, is a powerful strategy to optimize the gas sensing capabilities of graphene-based materials across various target gases. Table 2 summarizes some studies of graphene and its derivatives decorated with different metal nanoparticles for gas-sensing applications.

8.2.2. Graphene and its derivatives decorated with metal oxide nanoparticles

Numerous studies have explored the potential of graphene and its derivatives-based sensors decorated with metal oxide nanoparticles. Decorating graphene transistors with SnO₂ nanoparticles for a hydrogen gas sensor was investigated by Zhang et al. [167]. The sensor exhibited rapid response and recovery times (~1 s) at 50 °C for 100 ppm hydrogen. Analyses using XPS and AFM demonstrated that graphene's high carrier mobility combined with the minimal energy barrier at the interface with SnO₂ nanoparticles enables efficient charge transfer, resulting in enhanced sensitivity and selectivity for hydrogen detection. Gupta et al. [168] developed a copper oxide/reduced graphene oxide (CuO/rGO) hybrid nanostructure aimed at enhancing room-temperature gas sensing

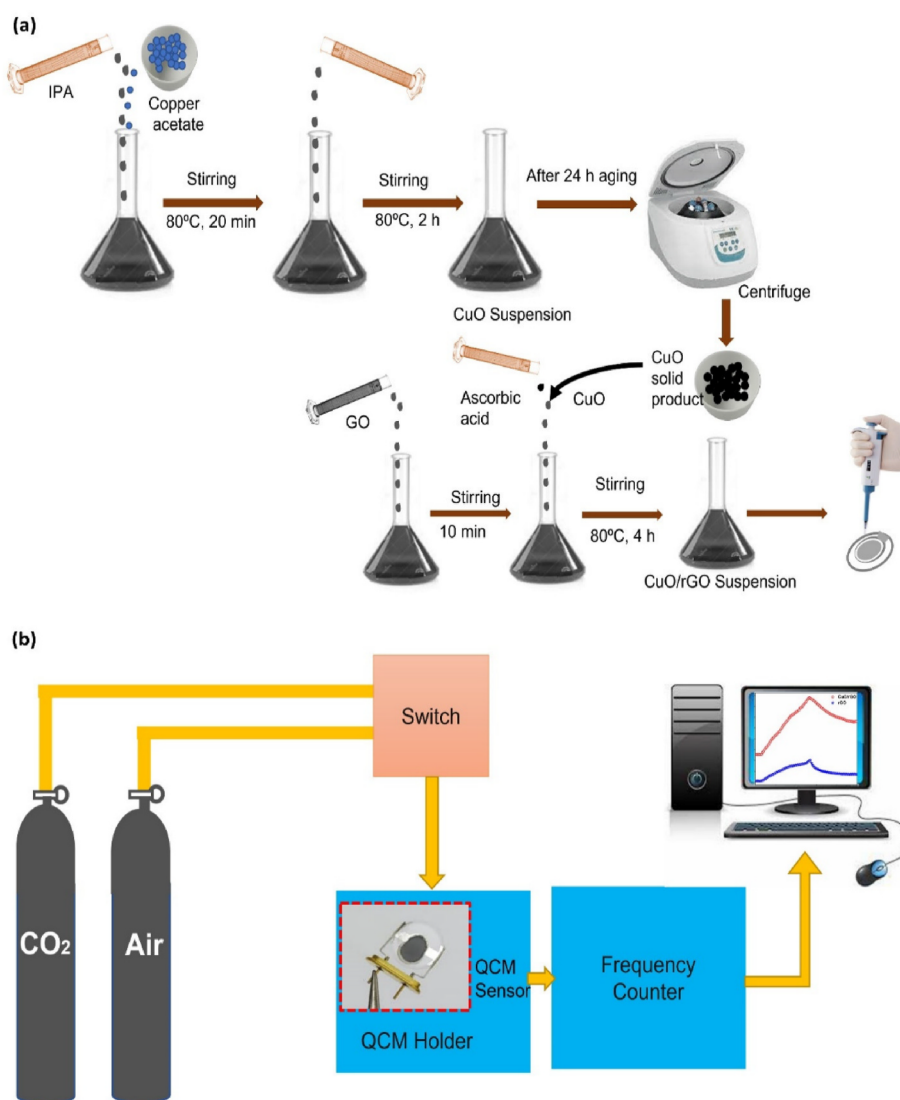


Fig. 11. Schematic diagrams of (a) synthesis of the CuO/rGO hybrid nanostructure and its sensor fabrication and (b) gas-sensing CuO/rGO hybrid nanostructure measurement set-up [170].

capabilities, particularly for CO_2 detection. The hybrid was synthesized by integrating CuO nanoparticles, derived from a copper acetate precursor, into graphene oxide during its reduction with ascorbic acid as shown in Fig. 11a. Characterization confirmed the successful formation of the hybrid material, revealing a high density of oxygen functional groups and uniformly distributed CuO nanoparticles ranging from 10 to 40 nm in size. When employed as a sensing layer in a quartz crystal microbalance (QCM) sensor as shown in Fig. 11b, the CuO/rGO hybrid demonstrated a significant improvement in CO_2 detection at room temperature, exhibiting a frequency shift of 438 Hz for 500 ppm CO_2 , which is more than double the response observed with rGO alone. The enhanced performance is attributed to the synergistic effect of CuO and rGO, where CuO nanoparticles provide active sites for gas adsorption, and rGO facilitates efficient charge transport. The gas-sensing mechanism involves the adsorption of oxygen molecules onto the CuO/rGO surface, forming oxygen ions that interact with CO_2 molecules, leading to changes in the charge carrier concentration at the hybrid surface and, consequently, the sensor's resistance.

Wang et al. [169] investigated the impact of microstructural variations on the NO_2 sensing performance of SnO_2 nanoparticles/reduced graphene oxide (SnO_2/rGO) hybrids at room temperature. They synthesized three distinct hybrids: SnO_2 NPs-RGO-PR via hydrothermal reduction, SnO_2 NPs-RGO-IS through one-pot hydrothermal synthesis, and SnO_2 NPs-RGO-SA by assembling SnO_2 nanoparticles onto rGO. Among these, the SnO_2 NPs-RGO-PR hybrid exhibited superior gas sensing characteristics, achieving a sensitivity of 65.5 % to 5 ppm NO_2 , with rapid response and recovery times of 12 and 17 s, respectively. The gas-sensing mechanism involves the adsorption of NO_2 molecules onto the sensor surface, leading to electron withdrawal from the conduction band, thereby increasing the sensor's resistance. In the case of TiO_2 nanoparticles, Ye et al. [170] presented a simple method for fabricating a TiO_2/rGO layered film sensor to detect ammonia at room temperature. The fabrication involved stepwise deposition of GO and TiO_2 layers, followed by a simple thermal treatment, resulting in a uniform TiO_2/rGO layered film. This approach resulted in a uniform TiO_2/rGO layered film, effectively preventing the re-agglomeration of graphene sheets and enhancing the interaction between graphene and TiO_2 nanoparticles. The fabricated sensor exhibited the enhanced response and excellent selectivity towards ammonia compared to pure rGO sensors as illustrated in Fig. 12, attributed to the synergistic effect between TiO_2 nanoparticles and rGO.

Srivastava et al. [171] developed graphene- WO_3 nanocomposite thin-film sensors for detecting NO_2 by drop-coating a dispersed solution of graphene and WO_3 onto alumina substrates. Transmission electron microscopy (TEM) confirmed a uniform distribution of WO_3 nanoparticles on the graphene sheets. Gas sensing measurements, conducted with varying graphene content (0.2, 0.5, and 1.0 wt %), showed that the graphene- WO_3 nanocomposite sensors exhibited nearly a threefold increase in response to NO_2 compared to pure WO_3 sensors at room temperature, with optimal performance observed at 250 °C. The enhanced NO_2 sensing performance of

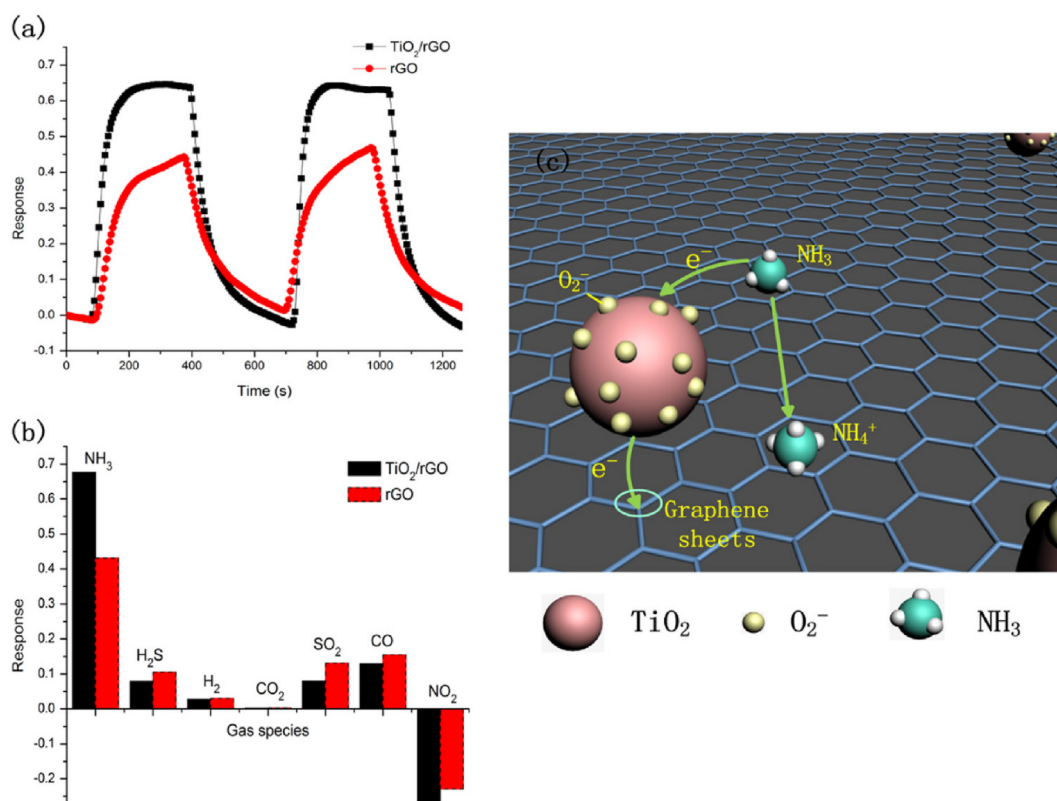


Fig. 12. (a) Response curves of pristine rGO and TiO_2/rGO sensors exposed to 10 ppm NH_3 ; (b) selectivity of the two sensors toward various gas species and (c) schematic illustration of the interaction between the sensing films and NH_3 molecules. Reproduced with permission from [172].

graphene- WO_3 nanocomposites is primarily attributed to changes in the Schottky potential barrier at inter-grain boundaries and the formation of n-p heterojunctions between n-type WO_3 and p-type graphene. Also, ZnO is widely used as a gas sensing material due to its high sensitivity, chemical stability, and low cost. Recent studies have reported on the use of ZnO nanoparticle-decorated graphene and its derivatives for gas-sensing applications. For instance, Wang et al. [172] investigated a sensing material prepared from a ZnO/graphene oxide nanoribbon (GONR) composite (ZnO/GONR) via a low-cost hydrothermal synthesis method for detecting NO_2 at room temperature. The hydrothermal process facilitated strong interfacial interactions between ZnO nanoparticles (20–60 nm diameters) and GONRs, enhancing electron transport and providing abundant active sites for gas adsorption. The composite sensor demonstrated improved performance over pure ZnO sensors, exhibiting a low detection limit of 1 ppm, rapid response and recovery times, and excellent repeatability when tested across NO_2 concentrations ranging from 1 to 50 ppm at room temperature. The enhanced sensing capabilities are due to the redox reaction between NO_2 molecules and oxygen anions adsorbed on the ZnO/GONR surface, which modulates the electrical resistance of the sensor.

As detailed in Table 2, we present a comprehensive summary of several studies and findings regarding the applications of graphene and its derivatives in combination with metal oxide nanoparticles for gas-sensing applications.

8.3. CNFs decorated with metal or metal oxide nanoparticles

CNFs are highly effective in gas-sensing applications due to their high surface area, excellent electrical conductivity, porous structure, and shortened pathways for electron transfer [180,181]. These features enhance the adsorption and interaction of gas molecules, as well as overall sensing performance. Additionally, the ability to adjust their morphology and easily functionalize them enhances selectivity and sensitivity to specific gases, while their lightweight nature enables use in portable devices [180,181]. Recent studies have explored the combination of CNFs with metal or metal oxide nanoparticles to improve gas-sensing capabilities. For example, Shooshtari et al. [182] improved ethanol gas sensing performance at room temperature by optimizing the density of vertically aligned carbon nanofibers (CNFs) and decorating them with Au nanoparticles. Using Plasma-Enhanced Chemical Vapor Deposition (PECVD), CNFs with varying densities were synthesized by adjusting the acetylene gas flow rates. Subsequently, Au nanoparticles were deposited onto the CNFs via aerosol impaction printing. Gas sensing measurements revealed that higher CNF densities led to increased sensor responses, with the highest density sample achieving a maximal responsivity of 10 %. Decoration with Au nanoparticles further enhanced the sensor's performance, showing a 300 % increase in response to 5 ppm ethanol compared to pristine CNFs. Fig. 13 (right) shows the presence of Au nanoparticles on the surface of CNFs with diameters less than 10 nm and the fabricated sensor (left). The enhanced response results from a greater surface area for gas adsorption and the catalytic activity of Au nanoparticles, which promote charge transfer between ethanol molecules and CNFs.

Zhang et al. [183] developed a flexible and responsive hydrogen sensor operating at room temperature. The sensor was fabricated by electrospinning polyacrylonitrile (PAN) to create a carbon nanofibrous mat, which was subsequently functionalized with amidoxime groups to facilitate the attachment of Pd nanoparticles. The prepared carbon nano-felt comprised randomly oriented nanofibers approximately 300 nm in diameter, uniformly decorated with Pd nanoparticles ranging from a few to tens of nanometers in size (Fig. 14a). Upon exposure to hydrogen gas, the sensor exhibited a decrease in electrical resistance (Fig. 14b), attributed to the catalytic dissociation of hydrogen molecules on the Pd surface and the subsequent formation of palladium hydride (PdH_x), which alters the

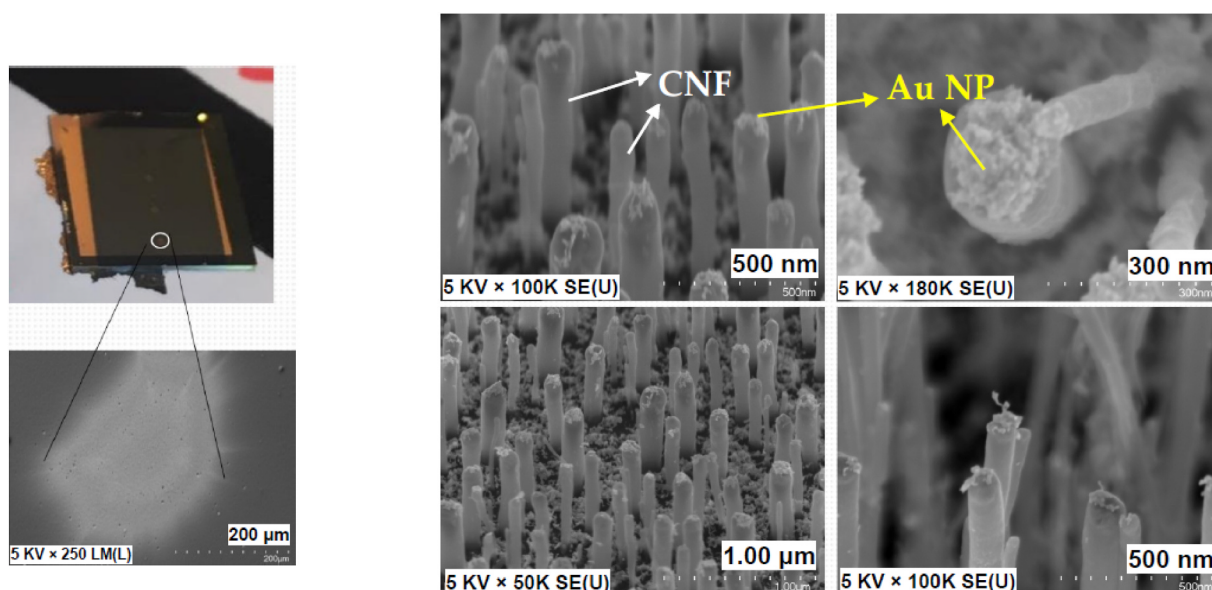


Fig. 13. Fabricated CNF sensor decorated with Au nanoparticles (left) and SEM images of the synthesized Au-CNFs (right) [184].

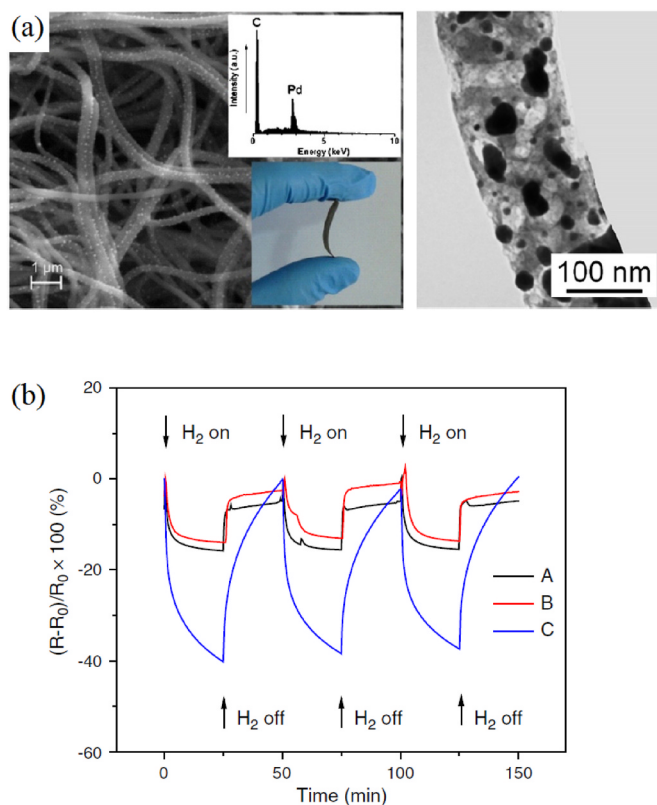


Fig. 14. (a) SEM (left) and TEM (right) images of CNFs decorated with Pd nanoparticles and (b) response curves upon exposure to H₂ at room temperature. Reproduced with permission from [185].

electronic properties of the Pd and the overall conductivity of the composite. This study highlights the efficacy of combining electrospun carbon nanofibers with metal nanoparticles to create sensitive and flexible gas sensors, and suggests that amidoxime-functionalized PAN mats can serve as versatile platforms for attaching various metal nanoparticles for diverse sensing applications.

Claramunt et al. [184] explored Au and Pd nanoparticles decorated on CNFs to enhance gas detection capabilities at room temperature. The prepared composite was deposited onto a flexible Kapton substrate, which had inkjet-printed interdigitated electrodes on

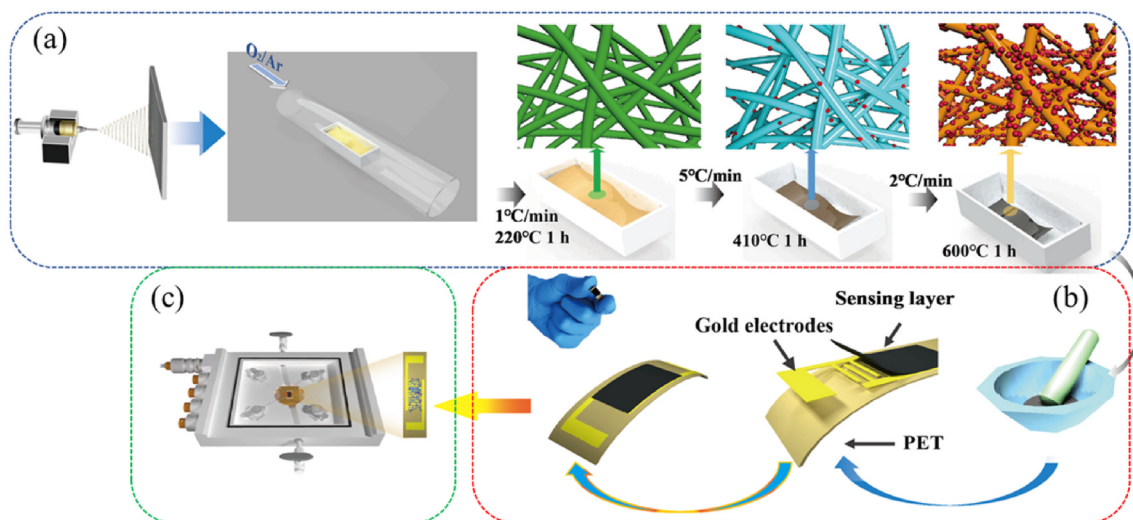


Fig. 15. Fabrication process of ZnO/CNF gas sensors. Reproduced with permission from [187].

one side and a heater on the backside. The decoration of Au and Pd nanoparticles produced varied responses to NH_3 and NO_2 gases. The sensing mechanism is based on (1) direct adsorption of gas molecules onto defects on the CNF sidewalls, which induces electron transfer and alters conductivity, or (2) adsorption of gas molecules onto the metal nanoparticles, leading to charge transfer between the nanoparticles and CNFs, resulting in changes in the electrical conductivity of the hybrid nanomaterial. The embedded heater allowed for controlled temperature conditions, enhancing the sensor's performance. The findings demonstrated that metal decoration of CNFs, combined with flexible substrate integration and embedded heating, presents a promising approach for developing high-performance, room-temperature gas sensors suitable for wearable and portable applications.

For decoration of metal oxide nanoparticles on CNFs, Fan et al. [185] fabricated ZnO nanoparticle-decorated carbon nanofibers (ZnO/CNF) through electrospinning of polyacrylonitrile (PAN) followed by sequential thermal treatments: preoxidation at 220 °C in air, oxidation at 410 °C, and carbonization at 600 °C in argon. Fig. 15 shows the process for fabricating ZnO/CNF gas sensors. The diameter of the synthesized ZnO/CNF was about 200 nm. The gas sensing test demonstrated that ZnO/CNF exhibited enhanced response and selectivity to NH_3 at room temperature compared to pure CNFs, significantly lowering the optimal operating temperature of pure ZnO sensors. The sensing mechanism relies on the interaction between NH_3 molecules and the oxygen species adsorbed on the surface of the ZnO nanoparticles. This interaction causes electrons to be released back into the conduction band of the ZnO resulting in a higher electron concentration. Consequently, the potential barrier of the p-n heterojunction is reduced, leading to a decrease in resistance. Moreover, CNFs provide a conductive network that facilitates efficient electron transfer and increases the surface area for gas adsorption. This synergistic effect between ZnO nanoparticles and CNFs enhances the sensor's sensitivity and selectivity.

Yan et al. [186] synthesized microporous heterostructure nanofibers of Sn– SnO_2 nanoparticles and CNFs using electrospinning, followed by heat treatment for ethanol sensing. The nanofibers comprised amorphous carbon interspersed with uniformly distributed Sn and SnO_2 nanoparticles. The results showed that these heterostructure nanofibers exhibited a twofold increase in response to ethanol at optimal operating temperatures, excellent selectivity, rapid recovery, and a linear response across 10–100 ppm concentrations. The sensing performance of the nanofibers for ethanol sensing arises from the high specific surface area and porosity provided by the CNFs, which facilitates gas diffusion and adsorption, and the synergistic interaction between Sn and SnO_2 nanoparticles, which enhances sensitivity and electron transfer during gas detection.

As examples of studies mentioned, the integration of CNFs with metal or metal oxide nanoparticles has emerged as a powerful strategy to enhance gas-sensing performance, particularly for applications requiring high sensitivity, selectivity, and room-temperature operation. CNFs offer a highly large surface area, porous structure, excellent electrical conductivity, and tunable morphology that collectively facilitate efficient gas adsorption, diffusion, and electron transfer. Decorating CNFs with noble metal nanoparticles like Au and Pd improves catalytic activity and promotes charge transfer mechanisms, as seen in enhanced ethanol and H_2 sensing. Similarly, decorating CNFs with metal oxide nanoparticles such as ZnO or SnO_2 introduces additional active sites and heterojunction effects that significantly boost sensor response and selectivity while lowering optimal operating temperatures. For instance, heterostructure nanofibers of Sn– SnO_2 /CNFs synthesized via electrospinning and thermal treatment demonstrated a twofold increase in ethanol sensing performance due to the synergistic effect of metallic and semiconducting phases embedded in a porous carbon matrix. These findings underscore the crucial role of CNFs as a conductive, high-surface-area platform for anchoring functional nanoparticles, enabling the development of high-performance, flexible, and portable gas sensors. Table 3 summarizes several studies of metal or metal oxide nanoparticles decorated CNFs for gas-sensing.

9. Conclusions and future prospects

This review highlights recent advancements in gas-sensing technologies through the integration of metal and metal oxide nanoparticles with carbon nanomaterials like CNTs, graphene, and CNFs. The intrinsic properties of carbon nanomaterials including their large surface area, superior electrical conductivity, and adjustable morphology, make them excellent platforms for gas detection. However, their sensing capabilities can be significantly enhanced through functionalization with metal or metal oxide nanoparticles. These nanoparticles contribute catalytic activity, increase the number of active sites for gas adsorption, and promote charge transfer, thereby enhancing sensitivity, selectivity, and accelerating response and recovery times. Various synthesis techniques such as hydrothermal, sol-gel, CVD, and microwave-assisted methods have enabled the creation of diverse nanocomposites tailored for specific gas detection. Additionally, the formation of heterojunctions at the interfaces between carbon nanomaterials and nanoparticles plays a crucial role in modulating the sensing mechanism, particularly through p-n junction and Schottky junction effects. Applications of these hybrid materials extend to the detection of hazardous gases such as NO_2 , NH_3 , H_2S , CO_2 , and ethanol at room or low operating temperatures, making them suitable for flexible, portable, and wearable sensing devices.

Despite significant advancements, several challenges need to be addressed before metal/metal oxide nanoparticle-decorated carbon nanomaterials can be widely utilized in real-world gas sensing applications. First, a key hurdle lies in achieving reproducibility and scalability in the synthesis of uniformly decorated carbon nanostructures with precise control over nanoparticle size, distribution, and surface chemistry. Although fabrication methods such as sol-gel, hydrothermal, and chemical vapor deposition show promise, scalable and cost-effective, manufacturing approaches are still urgently needed. Second, selectivity for specific gases in complex mixtures is still limited, as cross-sensitivity and environmental factors such as humidity and temperature significantly influence responses. Additionally, long-term stability and sensor degradation during continuous operation present practical barriers. Furthermore, integrating these nanomaterials into flexible, miniaturized, and low-power platforms requires careful optimization of device fabrication and signal-processing strategies. Future solutions may involve multi-component hybrid nanostructures, protective coatings, or the use of sensor arrays combined with machine learning-assisted signal processing or AI [188] to distinguish and identify complex gas mixtures under real environmental conditions.

Table 3

Applications of CNFs decorated with metal or metal oxide nanoparticles for gas-sensing.

Sensing materials	Target gases	Response/concentration	Operating temperature (°C)	Response/recovery times	Ref.
Au/CNF	ethanol	7 %/100 ppm	RT	60–90 s/35 s	[182]
Pd/CNF	H ₂	~500 sccm	RT	–/–	[183]
Au, Pd/CNF	NH ₃ , NO ₂	~500 ppm (NH ₃), 5 ppm (NO ₂)	RT	–/–	[184]
ZnO/CNF	NH ₃	12.3 %/50 ppm	RT	5 s/18 s	[185]
Sn–SnO ₂ /CNF	ethanol	~14/100 ppm	240	–/–	[186]
ZnO/CNF	H ₂	73.54 %/100 ppm	150	27.62 s/338.49 s	[187]

Moving forward, the trend toward flexible, wearable, and low-power sensors presents exciting opportunities for applications in environmental monitoring, industrial safety, and healthcare diagnostics. The integration of stretchable electrodes, flexible substrates, and self-powered systems through energy-harvesting mechanisms as well as IoT-based systems represents a promising frontier, moving these advanced sensing technologies closer to commercialization and real-world application. At the same time, a deeper understanding of fundamental sensing mechanisms at the atomic and molecular levels supported by in situ characterization, advanced spectroscopy, and computational modeling will be essential for rational sensor design. Moreover, the combination of advanced synthesis techniques with eco-friendly and cost-effective approaches will be essential for sustainable large-scale production. Finally, the integration of data analysis and AI will play a crucial role in realizing the full potential of next-generation gas sensors enabling enhanced selectivity, adaptability, and real-time monitoring under complex environmental conditions.

CRedit authorship contribution statement

Kriengkri Timsorn: Writing – original draft, Visualization, Resources, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Treenuch Ellis:** Formal analysis. **Yotsarayuth Seekaew:** Formal analysis. **Chatchawal Wongchoosuk:** Writing – review & editing, Validation, Supervision, Resources, Investigation, Funding acquisition, Formal analysis, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

This work was supported by the Thailand Science Research and Innovation, RDI PCRU, grant number 207965. In addition, C.W. gratefully acknowledges the Kasetsart University Research and Development Institute (KURDI) under grant number FF(KU)8.68 for any supports of this work.

Data availability

Data will be made available on request.

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