Graphene-Based Sensing Technologies for Gas, Biological, Thermal, And Humidity Applications

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Abstract. With the continuous development of technologies in various fields and the increasing demand for high-precision sensors, graphene and its composite materials have become popular in recent years because of their flat two-dimensional structure, extremely strong toughness, high thermal conductivity and remarkable electrical conductivity. This paper mainly introduces the physical and chemical properties of graphene, reviews the application and distinguished performance of graphene in efficient sensors from four aspects: gas sensors, biosensors, temperature sensors and humidity sensors in recent years, as well as briefly discusses the development prospects and bottlenecks of this material in combination with graphene materials and their sensors.

Keywords: graphene materials; highly sensitive sensors; graphene oxide; continuous monitoring.

1. Introduction

Graphene, with its unique two-dimensional structure and exceptional electrical, thermal, and mechanical properties, holds significant potential in cutting-edge technologies. The stable hexagonal structure of graphene molecules, high electron mobility, and high thermal conductivity make it suitable for highly sensitive sensors [1]. Besides, the graphene oxide (GO) and reduced graphene oxide (rGO) have various functional groups and active sites, which have the potential to combine with other substances to make up for some drawbacks of graphene [2].

Many sensor-based on graphene have been produced and show great performance. For gas sensors, the laser-induced graphene (LIG) gas sensor shows an obvious resistance change when it monitors monitored gas [3]. Metal phthalocyanine-doped graphene gas sensor exhibits great sensitivity to NO_2 [4,5][4]. For biosensors, the graphene electronic tattoo sensors are designed for continuous medical monitoring [6,7]. Graphene-modified enzyme liposome biosensor is able to detect heavy metals accurately [8]. The carboxyl-functionalized rGO/Ag Nanoclusters humidity sensor has a wider detection range and faster response, compared with traditional humidity sensors [9].

Based on properties of graphene and the sensors made of graphene, this paper further discusses the advantages and the challenges it faces, as well as analyzes the prospects of graphene. All in all, graphene is a material that has excellent performance and has great potential for future sensors, although there are some drawbacks, such as high cost and limitations in industrial production [1].

2. Basic Properties and Structure of Graphene and Graphene Composite Materials

2.1. Physical structure

Graphene is composed of one or two layers of carbon atoms, making it extremely thin with a thickness of only 0.335 nanometers. Coupled with its excellent electrical conductivity, graphene is suitable for making microsensors. The atoms in graphene form a regular hexagonal structure, which gives it high strength and toughness [1]. This property makes it less prone to deformation in real-world environments and ensures the accuracy of the sensor data [3].

2.2. Electrical properties/conductivity

The structure of graphene decides its high conductivity. Among the four valence electrons of a carbon atom in graphene, three electrons are bonded to other carbon atoms, while the remaining one can move freely within the graphene. At indoor temperature, the mobility of free electrons in graphene is 15,000 $cm^2/V \cdot s$ [1], which is ten times higher than that of silicon. Researchers from the University of Maryland stated that, after eliminating the influence of impurities, the mobility of free electrons within graphene will reach 200,000 $cm^2/V \cdot s$ [1]. It is precisely because of graphene's high electron mobility that the speed of internal electron movement is breakneck, reaching 1/300 of the speed of light, which is higher than that of traditional materials [1].

Unlike other materials, the electrical conductivity of graphene is less dependent on temperature change. Graphene has high electron mobility, fast electron movement speed, and low resistance, which lead to strong electrical conductivity, which is much better than copper and silver. Additionally, the lattice thermal vibration of graphene has a small effect on free electrons and is less likely to cause free electrons to collide with atoms and scatter, so its electrical conductivity is less influenced by temperature [1]

2.3. Mechanical Properties

Because of the extremely high bond energy of the carbon-carbon bonds in graphene, it possesses an exceptionally high tensile strength, reaching 120-180 GPa [1], which is more than 100 times that of steel. The hexagonal lattice of graphene makes its structure less prone to damage, demonstrating an extremely high hardness. Its hardness even surpasses diamond. Besides, Graphene has a very low density, with a surface density of 0.77 mg/m^2, a bulk density of 2.3 t/m^3 [1]. Besides, the low density and high strength make it have quite a high specific strength (strength value per unit mass). These features make the sensors made from it light, thin, and portable. Additionally, because of the high bond energy between carbon-carbon bonds, graphene not only has extremely high strength but also possesses excellent elastic deformation capacity, strong flexibility, and can be bent and folded, making it suitable for the production of flexible sensors [1].

2.4. Thermal Properties

Graphene has extremely high thermal conductivity. The thermal conductivity of single-layer graphene is about 5300 $W \cdot m^{-1} \cdot K^{-1}$ [1], as Table 1. shows, which is 10 times that of copper, 3 times that of diamond, and has exceeded many traditional materials [1]. However, its purity can influence the thermal conductivity of graphene. After doping with 1.1% of the carbon isotope ^{13}C, the thermal conductivity of graphene at normal temperature decreased by 10% to 15% [1]. In addition, as a two-dimensional material, graphene shows excellent thermal conductivity in the plane, but its thermal conductivity is relatively poor in the vertical direction. To improve its vertical thermal conductivity, researchers designed a three-dimensional material that combined graphene with carbon nanotubes in the vertical direction. Moreover, because of the strong carbon-carbon bond energy within the graphene, the thermal expansion of it is also very weak. When the temperature changes, its volume and shape do not change much.

Table 1. Thermal conductivity of several common materials [1].

Materials	Silver	Copper	Gold	Aluminum	Thermal conductive Graphene sheet	Carbon Nano tube	Dimond	Graphene
Thermal Conductivity/[W/(m·K)]	429	401	317	237	1500~1700	3000~3500	1000~2200	4000~6600

2.5. The Characteristics of Some Graphene Composites/Adaptability in Biosensors

2.5.1 Laser-induced graphene (LIG)

LIG is a kind of composite material which is produced by laser patterning on the polyimide substrates. It is a kind of porous graphene with a crystallite size in the tens of nanometers. This feature promotes its high sensitivity in detecting tiny molecules. With the different line density of the laser and the laser power, the van der Pauw structure created by the laser will let the sensor collect the compounds in the air well. It is suitable for making gas sensors. Besides, due to the fast-producing process and its simple structure, LIG has been widely used in medical and chemical detection [3].

2.5.2 GO/rGO

Graphene oxide has a large number of oxygen-containing functional groups (Epoxy, hydroxyl, carboxyl, and carbonyl groups, etc [2].) on its surface and edges, which makes it chemically active. It also features a large surface area, strong thermal conductivity, high light transmittance, and good electrical conductivity [10].

Reduced graphene oxide is based on GO. By reducing GO, rGO partially restores the graphene lattice and removes some oxygen functionalities, which enhance its conductivity and thermal stability, but it still retains a certain degree of machinability, which can combine with other materials [10]. There are three main methods [2], as Fig. 1 shows, which can reduce the GO, chemical, thermal, and optional methods, respectively. Although it is impossible to remove all the oxygen functional groups, rGO produced in different approaches exhibits different physical and chemical properties as well, and the degree of reduction has an influence on the performance of sensors. Besides, it has a layer-like structure of graphite, but the basal plane of rGO possesses several structural defects, containing oxidized functional groups, which result in providing more active sites for chemical detection. These features show rGO has the potential to make sensors and be applied in many fields. Furthermore, the cost of rGO is lower than pure graphene, which makes it possible to produce the rGO-made sensors on a large scale [1].

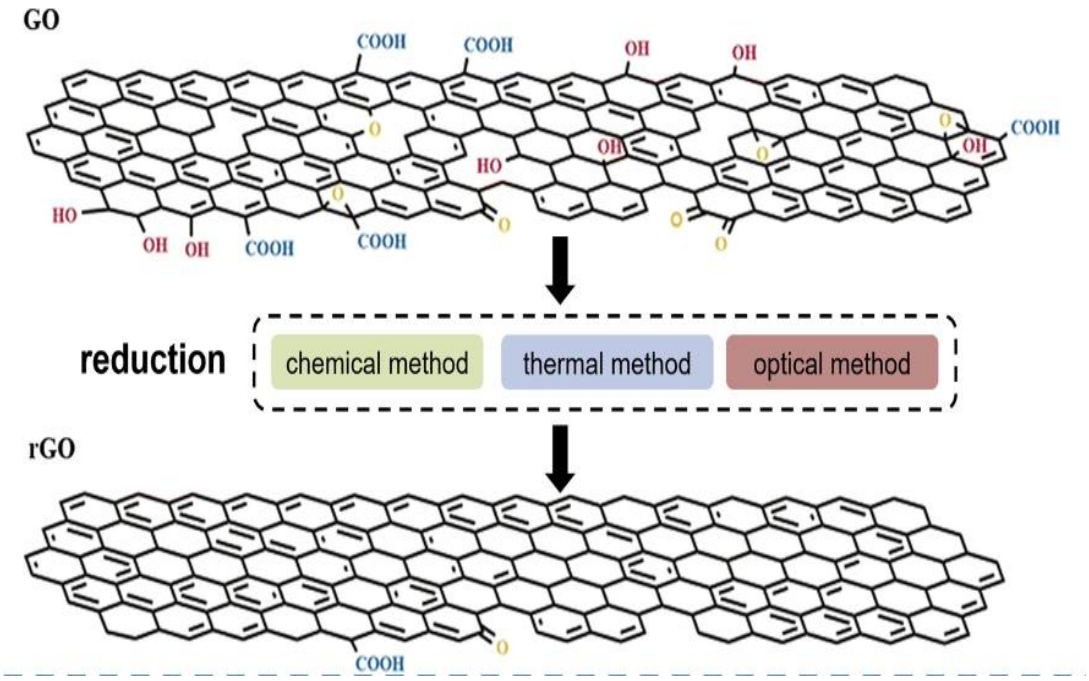


Fig. 1 Process of reducing GO [3].

3. Sensors based on graphene and graphene composite materials

3.1. Chemical and biological sensors

3.1.1 (LIG) gas sensors

LIG represents a three-dimensional porous graphitic material characterized by an extensive surface area, cost-effectiveness, and straightforward processing methodology, rendering it particularly suitable for small molecule detection. Through laser irradiation of carbon-rich precursors, LIG undergoes a photochemical and thermal transformation into graphene. Furthermore, the rapid evolution of gaseous byproducts during this process generates structural defects within the graphene lattice. These defects facilitate electron capture within the carbon rings while simultaneously establishing an ordered porous morphology, thereby augmenting the surface area, creating active sites, and enhancing catalytic performance. The carboxylic groups with the unpaired electrons which locate at the edges of the porous structure allow for sites to trap molecules, which make it sensitive to an organic compound, Volatile Organic Compounds (VOCs) [5], emitted by Artemisia species, which has been proven to be critical for understanding how the change of the local conditions play a role in species interactions in order to provide more detailed information for environmental conservation [3].

The methods of producing LIG utilize the laser and the properties of polyimide in order to achieve the accurate carving of graphene. Polyimide substrates can be transformed into laser-induced graphene through irradiation with a 450 nm laser [3]. Polyimide has significant absorption characteristics in the ultraviolet region, allowing different laser powers to achieve carbonization of carbon-rich materials. When spin-coating polyimide tapes and films on silicon substrates, experiments were conducted by adjusting the laser power and focusing distance. Finally, atomic jet printing (AJP) technology was used to print nano-particle silver ink to form electrode functions on the laser-induced graphene sensor.

After the bend testing and the VOCs response testing, which observe its resistance change (vital indicators for detection), the material shows great mechanical flexibility and VOCs sensitivity. The resistance of the tested films remains within 10% of the normalized resistance after experiencing 100 cycles with a bending radius of 15 mm [3]. Additionally, the Roman spectra also exhibit the structural integrity of LIG. A similar study taken in a parallel direction has the same results as the former. The mechanical test suggests that the LIG is suitable for a real-world environment. Furthermore, the VOCs responding test shows its excellent performance as well. The most visible LIG sensor responds to almost 5% resistance variation as well as consistent recovery when cycled over 12 min [3]. Simultaneously, researchers also discovered that the shapes of materials will affect the resistance variation. Among all the tested LIG sensors, the serpentine-patterned LIG on Kaptone performs best. These data illustrate that it has a good effect in real-time environmental detection. Expect the mechanical flexibility and the sensitivity, the low cost of LIG, and the wide selectivity of different gases to make it possible to be produced on a large scale and respond to various gases such as helium, oxygen [3].

3.1.2 Metal phthalocyanine-doped graphene gas sensors

Graphene has a single layer of carbon atoms arranged in a hexagonal lattice, which makes it one of the most promising 2D materials for future gas sensors. However, it is facing a challenge of slow recovery time, often needing annealing at a relatively high temperature to fully restore its abilities [5]. In order to improve its selectivity and sensitivity, the research focus has shifted from pristine graphene to its chemically and physically modified derivatives.

To solve the problem, interfacing graphene with organic molecules can shift graphene from neutral into either a p-type doped (positively charged) or an n-type doped (negatively charged) state via net charge, which is a great solution. Within the framework, mental phthalocyanine (MPcs), organic macrocyclic molecules, exhibit tunable chemical, electronic, and optional properties, making them suitable for applications in gas sensors. In this research, researchers focus on Fe, Co, Ni, and Cu as central metal cations, as well as using NH_3 and NO_2 as model gases [5]

Through density functional theory calculations [5], the electron charge transfer from NH_3 or NO_2 to the graphene layer is facilitated by the metal d_z2 orbital of the MPc molecule via its hybridization with the frontier molecular orbitals of the gases: specifically, HOMO of NH_3 and LUMO of NO_2. Regarding NH_3, charge transfer exclusively occurs under p-type doping conditions. In all cases, FePc consistently demonstrates the highest sensitivity, whereas the performance of CoPc, NiPc, and CuPc exhibits a decreasing trend. For NO_2, under both neutral and n-type doping conditions, sensitivity enhancement is observed, with the maximum response detected in n-doped FePc/Gr and CoPc/Gr systems.

3.2. Biological Sensors

3.2.1 Graphene Electronic tattoo sensors

Traditional medical sensors are facing drawbacks like large thickness, high cost, and are unable to continuously monitor [7]. However, graphene, a 2-dimensional material, has high electrical and thermal conductivity, which suggests it is suitable for producing lightweight, sustainable monitoring flexible biological monitoring sensors [6,7]. The graphene electrical tattoo (GET) is engineered with filamentary serpentine structures and fabricated through an efficient "wet transfer, dry patterning" methodology that optimizes both cost and time. Fig. 2 specifically introduces the process of producing the GET [7].

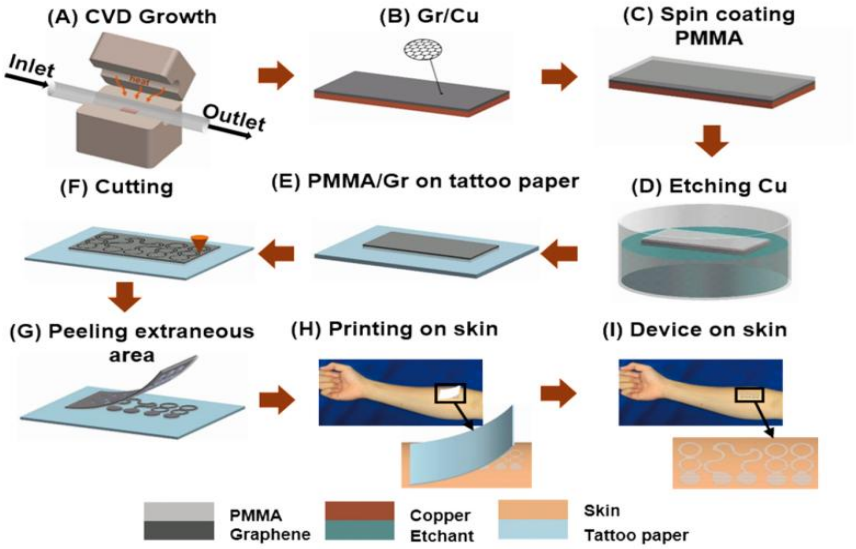


Fig. 2 (A, B) An atmospheric pressure chemical vapor deposition system (APCVD) was used to produce graphene on the copper foil. (C) Combine Less than 500 nm thick PMMA with graphene vortically. (D) Copper was moved away (E). The Graphene/PMMA (Gr/PMMA) was precisely transferred onto tattoo paper, with the graphene layer oriented upwards. (F) Gr/PMMA was clipped to specific shape. (G) Excrescent Gr/PMMA was moved away from the tattoo paper and the GET was thus produced. (H) Applying GET on the skin as a temporary tattoo and taking it away after a while. (I). The eventual state of GET on the skin [7].

After various tests, researchers found many benefits of GET. Compared with traditional technologies for electrophysiological measurements that use heavy flat plate electrodes, GET is 463 ± 30 nm thick, 85% transparent, and can elongate over 40% [7]. It integrates seamlessly onto human skin like a temporary tattoo, conforming perfectly to the skin's microscopic topography via van der Waals interactions. Compared with gold, a biocompatible and non-irritating material, always the top choice for dry electrodes and interconnection materials in electronic tattoos, graphene is cheaper, which is suitable for making temporary medical testing.

3.2.2 Graphene-modified enzyme liposome biosensor

The electrochemical sensing detection method for heavy metals has the advantages of simple operation and rapid response. However, most of the electrochemical sensors for heavy metal detection are based on core active enzymes, and this type of sensor has limitations in sensitivity and stability of the active enzyme. To improve the sensitivity of the detection system, this study chose graphene oxide as the electrode modification material and used the film evaporation-freeze-thaw combined method to construct a semi-closed glucose oxidase liposome reactor with a "cellular" permeable structure to enhance the stability of glucose oxidase. Nanoscale graphene was prepared by the modified Hummers method, and self-assembly technology was used to combine it with the glucose oxidase liposome reactor, with the aim of constructing a new type of glucose oxidase electrochemical biosensor for the detection of heavy metal residues (Hg^{2+} and Cu^{2+}) [8].

Compared with the cyclic voltammetry curve of the unmodified graphene working electrode, the biosensor working electrode modified with graphene shows higher redox peaks, indicating that the modification of graphene with a high specific surface area accelerates the migration efficiency on the surface of the working electrode, increases the reaction degree, and improves the detection sensitivity [8].

Under the optimal conditions, the electrochemical responses of the enzyme liposome biosensors modified with graphene to different concentrations of Hg^{2+} and Cu^{2+} were measured. Obvious oxidation peak currents were observed when detecting solutions of different concentrations of heavy metal ions. The oxidation peak of Hg^{2+} appeared at 0.15 V, while that of Cu^{2+} occurred at 0.20 V. Through regression equation calculation, the detection limit of Hg^{2+} was 10.97 ng/mL when the detection concentration was 10^{-10} to 10^{-5} mmol/L, and 3.60 ng/mL when the detection concentration was 10^{-5} to 10^{-2} mmol/L; the detection limit of Cu^{2+} was 87.70 ng/mL within the range of 10^{-10} to 10^{-2} mmol/L [8]. The lowest detection limits obtained by this method were significantly lower than the national standards.

3.3. Temperature Sensors

3.3.1 Wearable Temperature Sensors Based on Reduced Graphene Oxide Film

Body temperature constitutes a crucial physiological indicator. The structure of the temperature sensors plays a significant role in the accuracy of monitoring and user experimentation. Reduced graphene oxide (rGO) has been widely used in temperature sensing. rGO, derived from GO, shows excellent electrical conductivity (7.2×10^3 S/m) and thermal conductivity (5300 $W \cdot m^{-1} \cdot K^{-1}$) [1]. Its sensitive thermal response and high temperature coefficient of resistance make it ideal for temperature sensing.

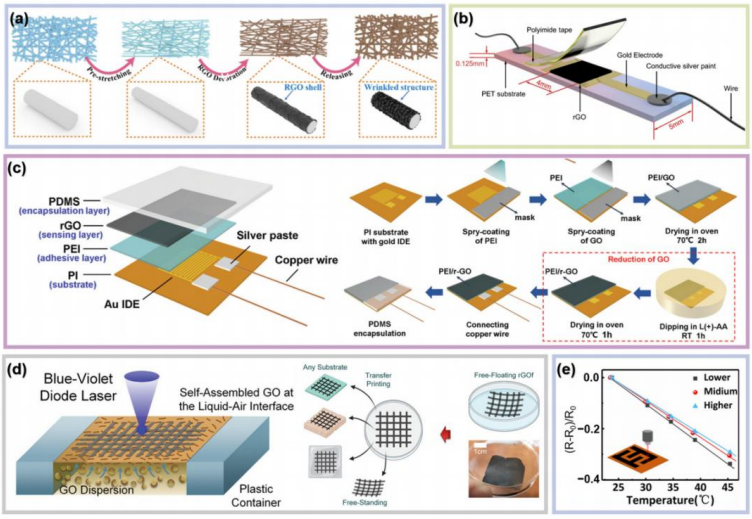


Fig. 3 (a) The processes of producing rGO temperature sensors; (b-e) the structure of temperature sensors, and some graphs of experimental data [11,12].

The Fig.3 shows the method of producing a pure rGO temperature sensor, and the basic structure of typical rGO temperature sensors. Fig.3 (a) shows the preparation of the rGO shell layer components on the surface of nanofibers. Fig.3(b) illustrates the structural configuration of the temperature sensor, the normalized resistance variation during thermal cycling (heating and cooling processes), and the normalized temperature response under various bending angles. Fig.3 (c) shows the multilayer structure of the temperature sensor and the fabrication procedure of the adhesive temperature sensor. Fig.3 (d) exhibits that the laser directly reduces the self-assembled GO sheet in order to manufacture rGOf at the solution surface. The reduced graphene oxide fabric (rGOf) forms a free-floating sheet, suitable as a self-supporting material or transfer-printable component on various substrates, regardless of their rigidity or flexibility. Fig.3 (e) The relative resistance variations as a function of temperature across diverse. The thermally activated charge carriers are the key points that decide the temperature dependence of rGO. The probability of charge carriers in the valence band moving to the conduction band increases or decreases with the variation of temperature [3].

3.3.2 Humidity Sensors

The measurement of humidity plays a vital role in many fields, such as medicine, food manufacturing, and so on. It is essential to enhance the accuracy of monitoring humidity. Although organic molecules modified with rGO have improved the response rate, selectivity and accuracy of sensing ability by utilizing various needed properties, it still has several drawbacks like poor hydrophilicity and poor recovery after water absorption. But Silver (Ag) can enhance more active sites, which are able to combine with water molecules, because of its active chemical properties. In this research, researchers tried to fabricate H3BTC-rGO/Ag nanocomposites via a facile one-step reduction method, which uses 1,3,5-benzenetricarboxylic acid (H3BTC) (as Fig.4 shows), GO, and silver nitrate as the substrates [9].

Using sodium hydroxide as the medium, GO and 1,3,5-benzenetricarboxylic acid (H3BTC) were mixed in deionized water. At this point, H3BTC adhered to the surface of graphene oxide through π–π interactions in a supramolecular assembly manner. Then, silver nitrate solution was added, and silver nanoparticles were uniformly attached to the pores of H3BTC-rGO sheet [9]

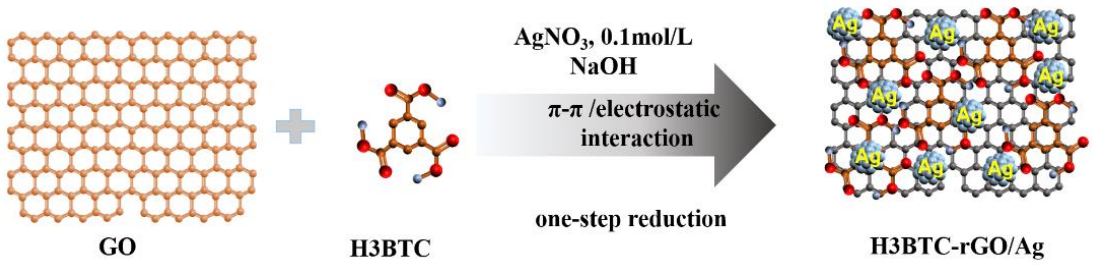


Fig.4 The principle of synthesizing H3BTC-rGO/Ag [9].

Compared with other humidity sensors, the H3BTC-rGO/Ag sensor responds faster, having higher sensitivity and a wider detection range, which indicates that H3BTC-rGO/Ag-0.25 is one of the most suitable materials for full humidity sensors. The hysteresis decides the efficiency of humidity sensors. The lower the hysteresis it has, the higher the efficiency it gets. The H3BTC-rGO/Ag-0.25-based sensor shows a hysteresis of less than ±5%RH, which is 2% less than the H3BTC-rGO-200-based sensor. [9].

3.4. Comprehensive Analysis: From Strengths to Future Prospects of Graphene

The graphene itself exhibits high electrical and thermal conductivity, which makes it suitable for highly sensitive sensors. Besides, due to its 2D structure, the sensors made of graphene are much thinner than others. The graphene composite materials, like GO, rGO, have various functional groups, such as hydroxyls, carboxyls, enhancing the conductivity, adding more active sites, as well as increasing their specific surface area. Furthermore, the cost of GO and rGO is lower than that of pure graphene, which makes it possible to produce them on a large scale [1]. For gas sensors, because of

the high electrical conductivity, when it senses the gas to be measured, its resistance changes particularly significantly [3]. For biosensors, their biocompatibility and two-dimensional structure give them an important advantage in flexible continuous monitoring biosensors [6,7,8]. For temperature sensors, because of their high sensitivity and compact design, it has been applied in many fields like monitoring respiration, finger touch, and so on [3,11,12]. For humidity sensors, rGO can better interact with other substances, making up its hydrophobic deficiency, and thus demonstrating a wider measurement range and more accurate measurement precision [9].

To make graphene play its role, its quality must meet the requirements. The applications mentioned in the previous chapters are all based on the premise of ideal high-quality graphene. In the laboratory, researchers can prepare high-quality graphene by using high-quality raw materials and strictly controlling the preparation process. However, in industrial production, these conditions are difficult to meet, so the quality of the produced graphene is not ideal.

At present, the preparation of graphene generally requires professional equipment and complex processes, which makes the preparation cost of graphene quite high and its price expensive, usually reaching several hundred yuan per gram, comparable to that of gold [1]. However, the performance improvements it brings cannot make up for the cost, that is to say, the cost-performance of graphene products is relatively low, and it is difficult to attract consumers. For example, it was reported that in 2013, a domestic enterprise in China put into production a graphene film production line with an annual output of 30,000 square meters, but the quality of the product was not ideal, and the production cost was high, resulting in very low sales [1].

Graphene is named for its excellent electrical and thermal conductivity and other advantages, and related research has made significant progress [13]. The research on graphene oxide and graphene composite materials has also developed rapidly. Besides people taking advantage of its outstanding performance, it also faces many problems and challenges, such as high cost, low cost-effectiveness, and a large gap between actual and ideal quality. However, it took more than 20 years for silicon materials to be applied after their discovery, and carbon fibers took over 50 years from discovery to large-scale application [1]. The bottlenecks and difficulties that graphene is currently facing may also be solved in the future. At the same time, this also inspires us to explore more "graphene-like" materials, such as layered molybdenum disulfide and two-dimensional black phosphorus. In addition, we can also explore the properties of compounds of reduced rGO, GO, and other materials to better utilize graphene [1].

4. Summary

To sum up, the data and applications show that graphene has great potential in highly sensitive sensors in many aspects. Although a lot of applications made of graphene have come out, there are still many challenges to deal with. Each of the main preparation methods for composite materials has its own drawbacks and shortcomings. To obtain a preparation method that is easy to control in operation, has low production costs, a high raw material utilization rate, and excellent product quality, further research and exploration are still needed. The combination of high electrical and thermal conductivity makes this material highly suitable for diverse sensor applications.

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