Research Progress of Gas Sensing Performance of 2D Hexagonal WO₃

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Metal oxide semiconductor gas sensing materials have attracted great research interest in the gas sensor field due to their outstanding physical and chemical properties, low cost, and easy preparation. Among them, two-dimensional hexagonal tungsten trioxide (2D h-WO₃) is especially interesting because of its high sensitivity and selectivity to some gases. We firstly introduce the characteristics of 2D h-WO₃ gas sensing materials and discuss the effects of microstructure, oxygen vacancy, and doping modification on the gas sensing properties of 2D h-WO₃ mainly. Finally, we explore the application of 2D h-WO₃ gas sensing materials and propose some research directions.

Keywords: 2D, hexagonal WO₃, gas sensing, oxygen vacancy, metal oxide semiconductor

INTRODUCTION

As a critical component of the intelligent detection system, the gas sensor (Lee et al., 2018) has been widely used in environmental monitoring (Ji et al., 2019a), respiratory analysis, explosive gases, and automobile exhaust detection. Based on different working mechanisms, the developed gas sensors include semiconductor gas sensors (Morrison, 1987a; Zhang et al., 2021), polymer gas sensors (Zee and Judy, 2001), and electrochemical gas sensors (Tierney and Kim, 1993). Among them, the semiconductor gas sensors can also be divided into resistive and non-resistive types, while the resistive semiconductor gas sensors have advantages of high sensitivity and easy preparation (Seiyama et al., 1962). Meanwhile, compared with carbon and other organic gas sensing materials, the resistive metal oxide gas sensors (Nazemi et al., 2019) have become the research hotspot due to their high responsivity (Demarne and Grisel, 1988) and excellent selectivity (Morrison, 1987b). As a highly sensitive metal oxide gas sensing material, tungsten trioxide (WO₃) has attracted extensive attention because of its unique physical and chemical properties (Salje and Viswanathan, 1975), and its applications in photocatalysis (Dong et al., 2017) and electrochromic (Adhikari and Sarkar, 2014).

WO₃ is a typical metal oxide semiconductor with various phase transition structures, while different phases can induce different gas sensitivity. The stable structures at room temperature are m-WO₃ and h-WO₃. In recent years, as the most stable structure, m-WO₃ has attracted much attention (Hübner et al., 2010; Oison et al., 2011), but bulk m-WO₃ gas sensors are not sensitive to some gases at 25°C–500°C, such as CO (Ahsan et al., 2012) and H₂S (Szilágyi et al., 2010). Therefore, it is urgent to improve the gas sensitivity of WO₃ at room temperature effectively. Xu et al. (2008) found that the sensitivity of h-WO₃ almost linearly increases with CO concentration at room temperature. Szilágyi et al. (2010) found that h-WO₃ becomes more sensitive than m-WO₃ compared to m-WO₃ when the concentration of H₂S is 10 ppm. Meanwhile, the large hexagonal and trigonal tunnel structures of h-WO₃ result in it having a high specific surface area (as shown in Figure 1) (Balaji et al., 2009), indicating that h-WO₃ is an excellent candidate material for gas sensors.
To effectively improve the gas sensitivity of the material, various preparation methods have been used to prepare WO₃ nanomaterials on various dimensions (0D, 1D, 2D, and 3D) (Qin et al., 2010; Zhang et al., 2010; D’Arienzo et al., 2014). Among them, 2D nanomaterials are widely used because of their high specific surface area and abundant oxygen vacancies (Yang et al., 2016; Liu et al., 2017; Yang et al., 2017). The unique characteristics of 2D WO₃ nanostructure compared with the bulk material include (1) higher specific surface area, which will provide more interaction area between tested gases and gas sensor surface molecules; (2) quantum confinement effects, due to the inherently small size of nanostructure materials, that can significantly affect charge transport, electronic band structure, and optical properties (Zheng et al., 2011). Based on this, we mainly review the effects of microstructure, oxygen vacancy, and doping modification on the gas sensing performance of 2D h-WO₃ and explore the application prospect of the 2D h-WO₃ gas sensor.

CHARACTERISTICS OF 2D h-WO₃ GAS SENSING MATERIAL

As a kind of metal oxide semiconductor, 2D h-WO₃ gas sensing material has been an excellent candidate material for gas sensors due to its advantages of easy preparation, stable crystal structure, high specific surface area, and abundant oxygen vacancies.

Easy Preparation

Table 1 shows some typical preparation methods of 2D h-WO₃. Among them, the hydrothermal method is the most widely used. According to this method (Kitagawa et al., 2009; Szilágyi et al., 2010; Ji et al., 2019b), (NH₄)₁₀W₁₂O₄₁·5H₂O is firstly put into a high-pressure cauldron as the raw material. Then, under high temperature and high pressure, (NH₄)₁₀W₁₂O₄₁·5H₂O recrystallizes to obtain precipitates (h-WO₃ crystals). Finally, the precipitates are removed and washed several times with deionized water to obtain the final product. Compared with vapor/liquid phase deposition methods, the hydrothermal method is simple and economical, and can prepare nanomaterials with high purity, good chemical uniformity and high dispersion. 2D h-WO₃ is classified as the surface-controlled gas sensor by a gas sensing mechanism.

Stable Crystal Structure

The phases of WO₃ can transform when it is annealed under different conditions. However, it does not simply form new nanostructures, but the original WO₆ octahedron distorts and twists to a certain extent and thus can form different crystal phases. The phase transition with temperature of WO₃ is shown in Figure 2 (Salje et al., 1997; Vogt et al., 1999; Roussel et al., 2000), which is monoclinic II (ε-WO₃ < −43°C) → triclinic (−43°C < σ-WO₃ < 17°C) → monoclinic I (17°C < m-WO₃ < 330°C) → orthorhombic (330°C < β-O₃ < 740°C) → tetragonal (740°C < α-WO₃). Meanwhile, Gerand et al. (1979) found that stable hexagonal WO₃ (h-WO₃) can be prepared by dehydration method at 200°C–400°C.

Tian et al. (2020) has calculated the gas (O₂) sensing on hexagonal WO₃ (001) surface by using the pseudopotentials method based on the density functional theory (DFT). The formation energy (E_form) of the h-WO₃ (001) monolayer is

| Structure | Materials | Method | Gas Type |
|-----------|-----------|--------|----------|
| 2D h-WO₃  | Nanosheet | Hydrothermal method | NH₃⁺ |
| 2D h-WO₃  | Nanosheet | Hydrothermal method | H₂S⁺ |
| 2D h-WO₃  | Film      | Hydrothermal method | NO₂⁻ |
| 2D h-WO₃  | Film      | Sol-gel polymerization | H₂⁺ |
| 2D h-WO₃  | Film      | Acidic precipitation | NH₃⁺ |
WO3 (Yamazoe et al., 2003), which implies that 2D h-WO3 may exhibit excellent gas sensing performance. For example, Tian et al. (2020) found that the conductivity and carrier concentration, and further affect the gas sensing performance of 2D h-WO3. SnP3 and GeP3, respectively. The surface oxygen density (Zhang, 2015) significantly improves the gas sensing performance of 2D h-WO3.

Effect of Oxygen Vacancy on Gas Sensing Performance of 2D h-WO3

In 1964, Kevane (1964) found that oxygen vacancies are easy to form in the preparation of metal oxide semiconductors. Makarov and Trontelj (1996) found that the oxygen vacancies would affect the conductivity, thus further affecting the gas sensing performance of WO3. However, the expression of oxygen vacancy on metal oxide semiconductor surfaces is not in agreement (Gillet et al., 2003). Until 2018, Tian et al. (2018) established a theory based on surface oxygen density (oxygen vacancies denoted as O-) for O-terminated, Vac O- for Vac O-terminated, WO- for WO-terminated, and Vac WO- for Vac WO-terminated, respectively. The surface oxygen densities are defined as \( d_o = 1, 1 > d_o > 0, d_o = 0, 0 > d_o > -1 \). Based on this, oxygen vacancies of the 2D h-WO3 surface can be expressed by surface oxygen density.

Recently, Tian et al. (2014) investigated the effect of oxygen vacancy on the gas sensing performance of CO on 2D h-WO3 (001) surface by using the first-principles calculations (Table 5). They found that the adsorption energy and charge transfer of CO of the defective O-terminated h-WO3 (001) surface decrease by

### TABLE 2 | The carrier mobility \( \mu \) at \( T = 300 \) K.

| Material                | \( \mu \) (10^3 cm^2 V^{-1} s^{-1}) |
|-------------------------|-------------------------------------|
| h-WO3 monolayer         | 0.886                               |
| Graphene                | 15.000                              |
| InP_3                     | 1.919                               |
| SnP_3                     | 7.150                               |
| GeP_3                     | 0.980                               |
| MoS_2                     | 0.201                               |
| 2D MoS_2 flake           | 0.600                               |
| SnO_2 bulk               | 0.160                               |
| WO_3 bulk                | 0.010                               |

The carrier mobility \( \mu \) calculated from the energy band is 886 cm^2 V^{-1} s^{-1} (as shown in Table 2) at \( T = 300 \) K. The value is higher than that of 2D GeP_3 (Gerand et al., 1979) and MoS_2 (Cai et al., 2014) and is about 88 times higher than that of bulk WO_3 (Yamazoe et al., 2003), which implies that 2D h-WO3 may have excellent gas sensing performance.

High Specific Surface Area

Sun et al. (2015) investigated the high surface area tunnels in 3D h-WO_3 by low-pressure CO_2 adsorption isotherms with nonlocal density functional theory fitting (NLDET), transmission electron microscopy (TEM), and thermal gravimetric analysis. They found that h-WO_3 has a large hexagonal tunnel structure (the diameter is 3.67 Å) and high specific surface area (45.585 m^2/g). Meanwhile, the large lateral size and ultrathin thickness of 2D materials provide it with ultrahigh specific surface areas and high ratios of exposed surface atoms (Zhang, 2015), significantly improving the gas sensing performance of 2D h-WO_3.

Abundant Oxygen Vacancies

The conduction band of 2D WO_3 mainly consists of W-5d electrons, and the valence band mainly consists of O-2p electrons. Chatten et al. (2005) found that abundant oxygen vacancies are related to the energy gap between O-2p and W-5d orbitals in non-stoichiometric tungsten oxide. Makarov and Trontelj (1996) pointed out that the oxygen vacancies in 2D WO_3 can affect the conductivity and carrier concentration, and further affect the gas sensing performance of WO_3. For example, Tian et al. (2020) found that oxygen vacancies provide electrons to O_2 gas molecules on the WO-terminated h-WO_3 (001) surface, thus effectively improving the gas sensing performance of h-WO_3 (001) surface to O_2.

Influencing Factors of 2D h-WO_3 on Gas Sensing Performance

When the gas sensors are exposed to the air, O_2 molecules are physically or chemically adsorbed on the surface of 2D h-WO_3.
0.68 eV and 0.002e, respectively, compared with the O-terminated h-WO3 (001) surface. For defective WO-terminated, the values of decrease are 0.4 eV and 0.011e, respectively. The result shows that the adsorption and sensing ability of CO on the defective O- and WO-terminated h-WO3 (001) surface decreases. The oxygen vacancy inhibits the oxidation reaction of reducing gas CO on the 2D h-WO3 (001) surface, which reduces the gas sensing performance of the 2D h-WO3.

Oxygen vacancy also inhibits the gas sensing performance of other reducing gases (H2S, CH4, H2) on the 2D h-WO3 (001) surface (Szilágyi et al., 2010; Tian et al., 2017; Wu et al., 2019) (Table 5). However, the inhibitory effect of oxygen vacancy on H2S and CH4 is unapparent. Although the gas sensing performance of H2S is inhibited by oxygen vacancy, the value (1.85 eV) is still large enough for effective adsorption of H2S on the surface. The adsorption sensing ability of CH4 on the 2D h-WO3 (001) surface is weak and the inhibition of oxygen vacancy makes it difficult to spontaneously adsorb on defective WO-terminated h-WO3 (001) surface. Moreover, oxygen vacancy has the strongest inhibitory effect on the gas sensing performance of H2 on the 2D h-WO3 (001) surface. The adsorption energy decreases from 2.62 to 0.16 eV and the charge transfer decreases from 0.635e to 0.065e. The gas adsorption ability of H2 on the 2D h-WO3 (001) surface greatly reduces with the decrease of surface oxygen density.

More recently, Tian et al. (2020) investigated the effect of oxygen vacancy on the gas sensing performance of O2 on the 2D h-WO3 (001) surface (Table 5) by the first principles with pseudopotentials method based on the DFT. They found that the adsorption energy of O2 of the defective O-terminated h-WO3 (001) surface increases by 0.05 eV and the charge transfer decreases by 0.104e compared with the O-terminated h-WO3 (001) surface. For the defective WO-terminated surface, the values of increase are 5.65 eV and 0.077e, relatively.

| TABLE 3 | Relationship between microstructure, particle size, and gas sensitivity of H2, NH3, H2S, and NO2 in h-WO3 (S is the detection scope, R is the responsiveness, and C is concentration). |
|---|---|---|---|---|---|---|
| Gas | Material | Size/nm | T/°C | S/ppm | R = Rg/Ra | C/ppm |
|---|---|---|---|---|---|---|
| H2 | Filma | 110–320 | 450 | 200 | 151.9 | 200 |
| | Nanoflowerb | 450–600 | 270 | 100 | 2.5–5 | 100 |
| | Nanospherec | 500–2000 | 250 | 10–80 | 0–5 | 80 |
| | Nanoparticlea | 50–100 | 300 | 10–50 | 5–5.5 | 50 |
| NH3 | Nanoroda | 30–100 | 400 | 50–200 | 22.5 | 200 |
| | Nanosheetf | 50–500 | 350 | 50–250 | 38.3 | 100 |
| | Nanoparticlea | 50–100 | 200 | 0–0 | 0 | 0–200 |
| H2S | Nanowired | 50–500 | 20 | 0–0 | 0 | 0–200 |
| | Nanosheetf | 50–500 | 300 | 0–40 | 45.86 | 40 |
| | Nanoparticlea | 700–1,000 | 75 | 1–10 | 5.8 | 10 |
| NO2 | Filma | 1,000–2000 | 200 | 0.01–0.5 | 103 | 0–0.1 |
| | Nanospherea | 500–2000 | 250 | 10–80 | 60–65 | 80 |

*aSone et al.(2018). bZhang et al.(2019). cWei et al.(2017). dWang et al.(2007). eSzilágyi et al.(2009). fJi et al.(2019b). gJi et al.(2019b). hSone et al.(2018). iJi et al.(2019b). jSone et al.(2018). kLiu et al.(2014). lShi et al.(2016). mSzilágyi et al.(2010). nMeng et al.(2015). oKitagawa et al.(2009). pZhang et al.(2019).
shows that the adsorption and sensing ability of O\textsubscript{2} are improved on the defective O- and WO-terminated h-WO\textsubscript{3} (001) surface. The oxygen vacancy activates the O-O bond of O\textsubscript{2} and promotes the reduction reaction of oxidizing gas O\textsubscript{2} on the 2D h-WO\textsubscript{3} (001) surface, which improves the gas sensing performance of the 2D h-WO\textsubscript{3}.

These results indicate that the effect of oxygen vacancy on gases with different redox properties is different. For reducing gases, the oxygen vacancy inhibits their oxidation reactions on the 2D h-WO\textsubscript{3} (001) surface and then reduces the gas sensing performance of the reducing gases. On the contrary, for oxidizing gases, the oxygen vacancy promotes the reduction reaction and then improves the gas sensing performance.

**Effect of Doping Modification on Gas Sensing Performance of 2D h-WO\textsubscript{3}**

Various methods have been performed to improve the gas sensing performance, to overcome the defects of pure metal oxides such as low sensitivity, low selectivity, and long response time for some gases (Liu et al., 2019). Among them, noble metal doping is one of the most common and effective methods. Due to the high electronic activity of noble metal elements, the activation energy of the reaction can be reduced during the contact reaction between the gas sensing material and the target gas, thus improving the gas sensing performance of the materials (Xu et al., 1990) when they react with target gases. Based on this, noble metals such as Au, Ag, Pd, and Pt are usually doped on WO\textsubscript{3} films to improve their sensitivity and selectivity to NO\textsubscript{x} (Penza et al., 1998; Chen and Tsang, 2003), H\textsubscript{2}S (Stankova et al., 2004; Hurtado-Aular et al., 2021), CH\textsubscript{3}COCH\textsubscript{3} (Feng et al., 2021), etc.

**SUMMARY AND PROSPECT**

The research progress of the gas sensing performance of 2D h-WO\textsubscript{3} has been reviewed. Firstly, we briefly summarize the
characteristics of 2D h-WO₃ gas sensing materials. Then, the effects of microstructure, oxygen vacancy, and doped metal on the performance of 2D h-WO₃ gas sensors are mainly discussed. We find that the 2D h-WO₃ gas sensor has better gas sensing performance than other WO₃ nanomaterials due to their small particle size and large specific surface area. Moreover, the effect of oxygen vacancy on the gas sensitivity of different oxidation-reducing gases on 2D h-WO₃ is different. Besides, we also note that noble metal doping can improve the gas sensing performance of 2D h-WO₃ due to the high electronic activity of noble metals and the reduction of reaction activation energy.

As we all know, 2D h-WO₃ is an excellent candidate material for metal oxide semiconductor gas sensors, which has vital research significance and wide application prospects in gas sensors. However, there are still some unsolved problems in 2D h-WO₃ that need to be completely solved, such as the low sensitivity and low selectivity to some gases. To solve the above problems, the possible solutions include the following: (1) Photoactivation method (i.e., activation of reactants by light), which can improve the sensitivity and selectivity effectively. Deng et al. (2012) activated mesoporous WO₃ sensing material and improved the sensitivity of WO₃ to HCHO by using visible light irradiation at room temperature. Moreover, Trawka et al. (2016) enhanced the sensitivity and selectivity of WO₃-based gas sensors greatly by ultraviolet irradiation. (2) Noble metal doping method improves sensitivity and selectivity. Adding precious metal catalysts has become an important method to improve the gas sensing performance of metal oxide semiconductors, because the catalyst has a great influence on the resistance and sensitivity of semiconductor gas sensing materials (Krebs and Grisel, 1993).

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All authors listed have made a substantial, direct, and intellectual contribution to the work, and approved it for publication.

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