



Improved Sensing Properties of Thermal Conductivity-Type CO₂ Gas Sensors by Loading Multi-Walled Carbon Nanotubes Into Nano-Al₂O₃ Powders

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Response time is the key index of on-line monitoring system. To improve the response speed of traditional bead thermal conductivity CO₂ sensor, this paper proposes to use multi-walled carbon nanotubes (MWCNTs) to improve the performance of gas sensor carrier. Nano-sized γ -Al₂O₃/CeO₂ powder was synthesized by chemical precipitation method under the action of ultrasonic wave. SEM morphology reveals a particle size of 20–50 nm. MWCNTs were hydroxylated and the solution was then prepared by adding a certain amount of dispersant under ultrasonic wave. The composite support of γ -Al₂O₃/CeO₂/MWCNTs was prepared by wet mixing carbon nanotube solution into the above support materials. Using dynamic resistance matching and black component technology, the influence of radiation heat and environmental temperature and humidity is reduced. Results show that the designed thermal conductivity sensor has consistent response and recovery time to different concentrations of CO₂, with a T₉₀ response time of 9 s and a T₉₀ recovery time of 13 s, which is faster compared to major commercial Carbon dioxide sensors. The average sensitivity of the sensor is 0.0075 V/10% CO₂. Therefore, the high thermal conductivity and pore characteristics of carbon nanotubes can effectively improve the response speed of the thermal conductivity sensor.

Keywords: gas sensor, thermal conductivity, CO₂, multi-walled carbon nanotubes, fast-response, nano γ -Al₂O₃

INTRODUCTION

A large number of greenhouse gas CO₂ emissions aggravate the global warming. Acid rain, haze, and other bad weather caused huge economic losses and serious environmental damage (Hansen and Sato, 2004). Controlling CO₂ emission is the fundamental way to deal with climate warming as generally agreed by the international community (Zhang et al., 2008). In industrial production, especially in coal mines, CO₂ is often a product of emissions, which is also a source of danger in the production process (there have been many carbon dioxide outburst accidents in the history of coal mining). Therefore, it is necessary to detect and control the CO₂ emissions in a distributed, real-time and accurate way (Ghosh et al., 2013; Zaitsev et al., 2017).

Thermal conductivity sensor is a thermal effect sensor that can measure the concentration of gas according to the difference of thermal conductivity of different gases and air (Gardner et al., 2020). Usually, the difference of thermal conductivity is converted into the change of resistance using the Wheatstone circuit. The heat conduction mode usually includes convection, conduction, and radiation that works at about 300°C. Its response mechanism determines that it has the advantages of large detection range, good working stability, and strong reliability. However, it also exhibits the problems of slow response time and low detection accuracy (Wei-Yong et al., 2006). To achieve high precision and fast response detection in a dangerous environment, it is necessary to improve the thermal conductivity and response sensitivity of the sensor.

In recent years, carbon nanotubes (CNTs) have been gradually used in the development of thermal conductive gas sensors because of their better thermal properties and pore structure compared to ordinary ceramic carriers (Huang et al., 2019). Researchers at home and abroad have studied gas sensors modified by carbon nanotubes (Zhang, 2012). The gas sensor fabricated by Guo et al. (2006) was coated with a small amount of MWCNTs between interdigital gold electrodes based on Al₂O₃ and has good gas sensing response to toluene at room temperature. Tang et al. (2020) used single-walled carbon nanotubes (SWNT) to fabricate NH₃ sensors, obtaining a superior sensitivity of 2.44% $\Delta R/R$ per ppm_v NH₃, which is more than 60 times higher than intrinsic SWNT-based sensors. Bin Shen (Shen et al., 2018) and others studied the pore forming technology of MWCNTs supported on aligned nanotubes and designed and fabricated a kind of hot-wire-coated ceramic powder thermal conductivity sensor for methane detection with a response recovery time of 8 s and 16 s (Xibo et al., 2013).

In this paper, MWCNTs are used to improve the blind hole carrier structure of traditional bead like thermal conductivity sensor, open up more gas transmission channels, and effectively improve the permeability of the sensitive material “ceramic bead” (Wu and Lin, 2006; Torres-Torres et al., 2013). The fabrication method and key technology of this kind of sensor are presented. The performance of the sensor is measured and the possible mechanism is discussed. Results indicate that the response and recovery time of the sensor by loading MWCNTs modification into nano-Al₂O₃ powders has been greatly shortened. The study has an important influence on the improvement of real-time detection technology of high concentration CO₂ in coal mines (Qin et al., 2011).

EXPERIMENTAL SECTION

Sensor Structure and Explosion-Proof Package Design

A thermal conductivity gas sensor consists of a detecting element and a compensating element, which are a pair of working components (Figure 1A) consisting of a platinum hot-wire resistor and carrier (Figure 1B). These two components are separately assembled into two standard tubes. One is non-sealing packaging (Figure 1C), and the other is sealing packaging, which

is used for the interference of humidity and the compensation of ambient temperature of sensitive component. A thermal conductivity sensor is then made by assembling the two components into the powder metallurgy housing (Figure 1D) to realize explosion-proof safety designing.

Manufacturing Process of the Sensor

The manufacturing methods of the sensitive element and the compensation element of the thermal conductivity sensor are similar, but the packaging form is different, which plays a role of differential compensation (Xue et al., 2013). The key factors that restrict the performance of the sensor are mainly the composition of the sensor carrier, microstructure, types and distribution forms of catalysts. The sensor's main manufacturing process consists of 10 steps such as carrier produced, carrier modification (mixed carbon nanotube and catalyst loading), element coil winding, coating carrier on the coil, element sintering, element blackening, element matching and element packaging, sensors aging and performance testing and so on. The specific process is shown in Figure 2.

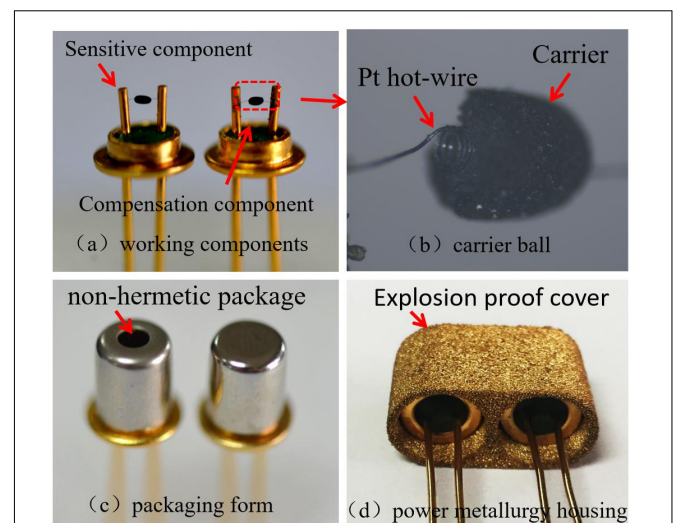


FIGURE 1 | Sensor assembly package. Label (a) shows working components, label (b) shows carrier ball, label (c) shows packaging form, label (d) shows powder metallurgy housing.

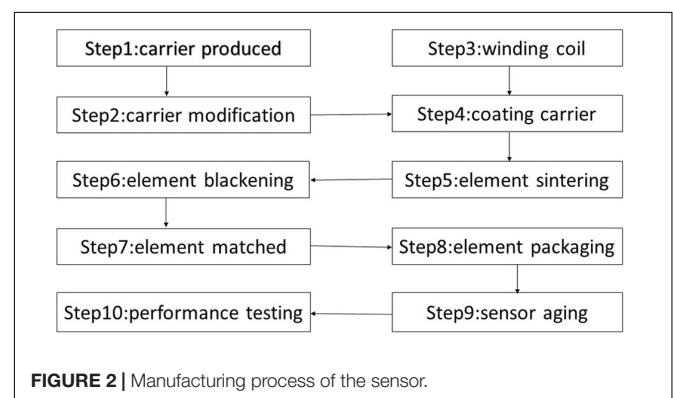
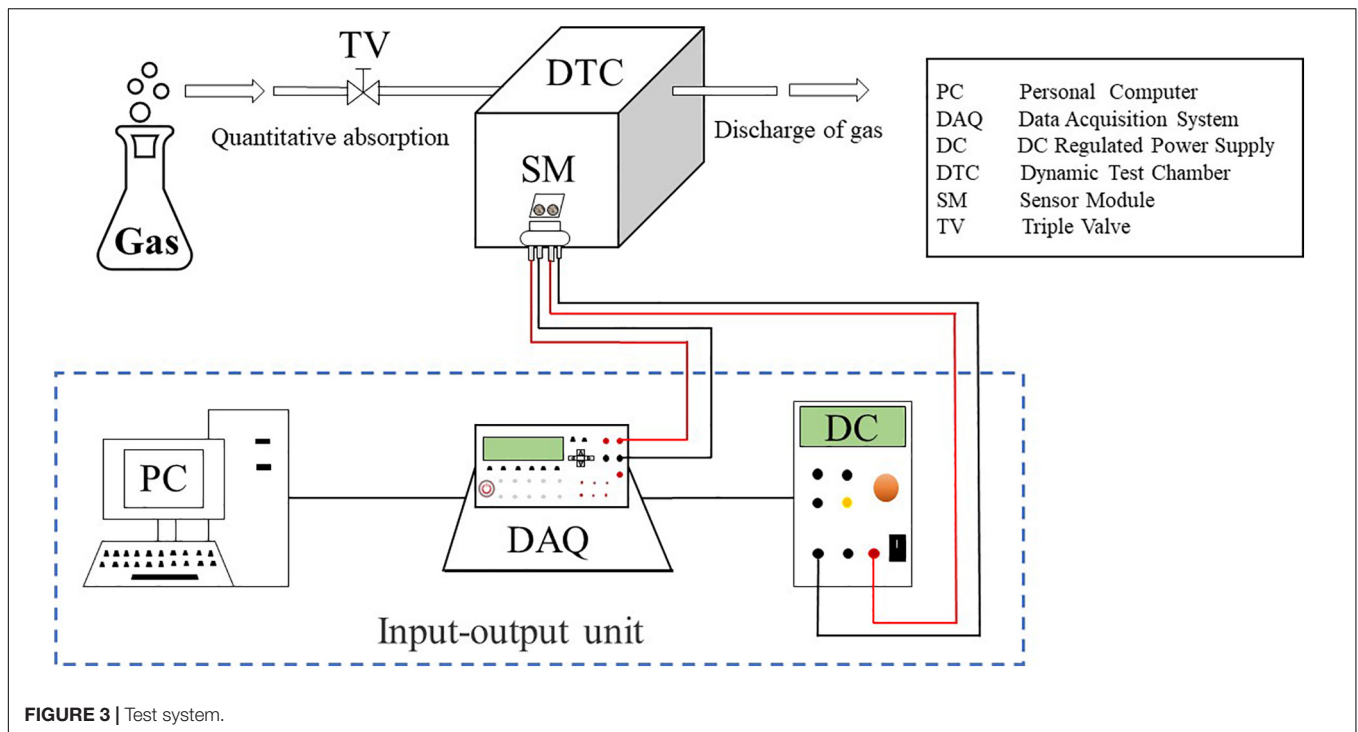


FIGURE 2 | Manufacturing process of the sensor.



Key Process Design

Composite Carrier Material Design

γ -Al₂O₃ nanoparticles have the advantage of a higher specific surface area. After being mixed with MWCNT, a large number of micro-nanopores will be formed (Zhang et al., 2018). Some of the through holes are conducive to the production of sensitive units for thermal conductivity sensors (Wu et al., 2013).

In this paper, nanoscale γ -Al₂O₃ ceramic ultrafine powder carrier materials were prepared by chemical precipitation method (Saha et al., 2005). Under the continuous action of ultrasonic wave, the solution composed of 0.02 mol formic acid, 5.4 g water and 7 ml isopropanol was slowly dropped into the mixture of 20.4 g isopropyl aluminium and 200 ml toluene. After completing the operation, the mechanical stirrer is started and the solution is heated to 50~60°C. The reaction is then

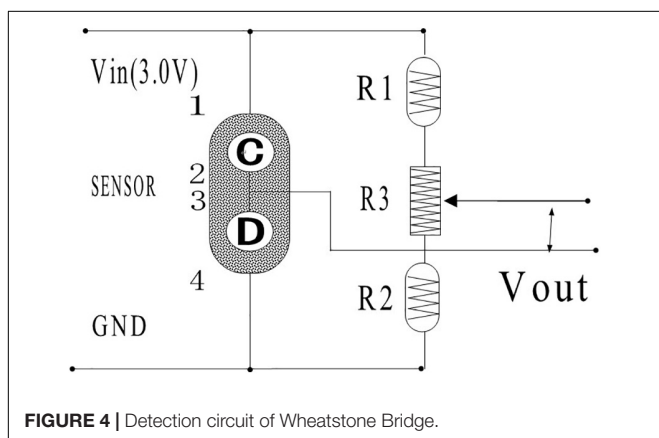
maintained for 1 h and then the semitransparent gel is obtained. The products were filtered, dried at 60°C for 12 h and dried again at 120°C for 1 h. The loose dry gel powder was then obtained. The nano-Al₂O₃ powder can be obtained by calcining the xerogel powder for 2 h at 700~800°C. It should be noted that the micro droplet adding mode under ultrasonic wave and calcination temperature are the key to the formation of nano-sized γ -Al₂O₃ powder.

Since MWCNTs are difficult to dissolve in water, hydroxylated MWCNTs (SSA > 490m²/g, Purity > 95wt%, Chengdu Organic Chemicals Co. Ltd) was selected in the experiment. SEM characterization showed that the MWCNTs had an outer diameter of 8 nm and a length of 5~30 μ m. To improve the dispersion uniformity of MWCNTs in Al₂O₃ powder, it is necessary to prepare MWCNTs in an aqueous solution by adding certain amount of dispersant under the action of ultrasonic wave.

To improve the thermal stability of the carrier, the target composite carrier also needs to be modified by adding 5% w/W Nano-CeO₂ powder (30~50 nm, 99.99% purity, Macklin). Finally, the material was added into γ -Al₂O₃/CeO₂ powder at the ratio of 5% w/W to form nano- γ -Al₂O₃/CeO₂/MWCNTs composite catalyst support.

Blackening of Sensitive and Compensation Components

Due to the high specific surface area and surface activity of nano- γ -Al₂O₃, the carrier made of it has strong adsorption to polar molecules (including water molecules in the air) (Liu et al., 2011), but the desorption effect becomes poor. At the same time, the grayscale of ceramic fired by the carrier γ -Al₂O₃/CeO₂/MWCNTs is low, which causes the heat radiation



effect to be aggravated, thereby affecting the detection of thermal convection effect.

In this paper, Pd and Pt particles are formed by impregnating pure chlorinated palladium acid solution ($\text{H}_2\text{PtCl}_6 \cdot 6\text{H}_2\text{O}$ and $\text{H}_2\text{PdCl}_6 \cdot 6\text{H}_2\text{O}$ analytically pure, Sinopharm Chemical Reagent Co., Ltd), which consequently electrothermally decompose the Pd and Pt particles to make the carrier change from gray to black. The heat dissipation efficiency of the carrier can be effectively reduced when the sensor is working. To reduce the catalytic effect of nano-Pd/Pt particles, lead nitrate solution was impregnated on the surface of black Pd/Pt particles and decomposed at high temperature to form desensitized lead monoxide. Finally, the sensitivity and compensation elements of thermal conductivity sensor without catalytic effect were formed.

Resistance Sintering and Thermal Decomposition

Three key steps must be completed to solve the problem of carbon nanotubes easily burning at high temperatures.

The first step of electrified sintering is sintering the carrier powder size at high temperature to combine the grains and make micro/nano-holes, so that the carrier has a certain mechanical strength and a stable high specific surface area. The sensitive

coil of the coated carrier was loaded with DC voltage under the protection of high-purity nitrogen and around 550°C, and the sintering current of 150 mA was maintained for 60 min.

Second, under the protection of high-purity nitrogen, a sintering current of 140 mA is passed through at about 500°C, and the current is maintained for 30 min to thermally decompose the palladium chloride solution impregnated in the carrier. Therefore, the sensitive components and compensation components will turn black.

The third step is in high purity nitrogen protection and under 550°C, through sintering current of 150 mA, decompose the dipping lead nitrate solution black carrier 30 min, due to the thermal decomposition of lead nitrate solution to form the oxide and PbO, will eliminate the Pt and Pd catalyst carrier. As a result, the sensitive component will only have the thermal conductivity effect during working.

Test System Building and Performance Testing Method

The dynamic test system in Figure 3 consists of the input and output devices (including computer, DC stabilized power

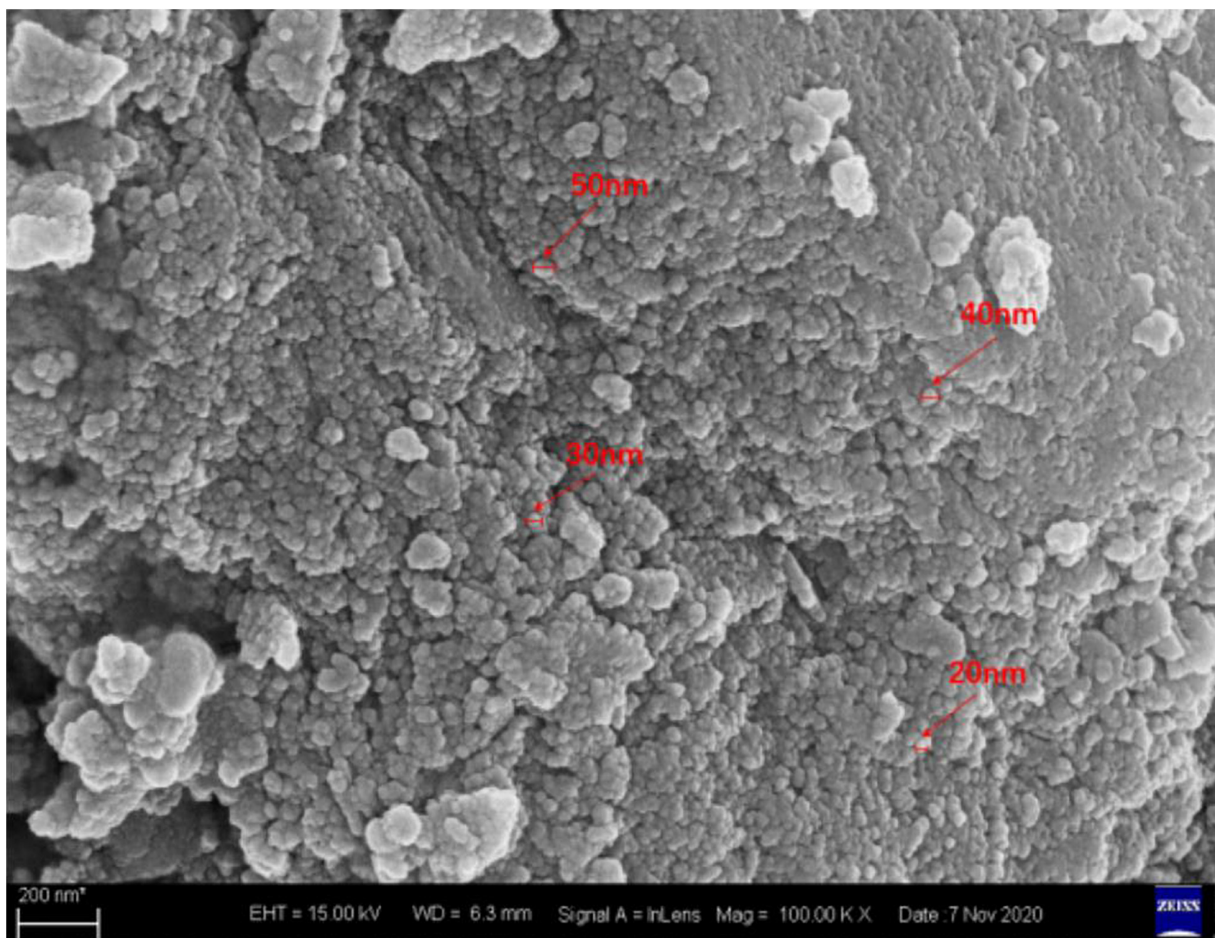


FIGURE 5 | SEM image of the Al_2O_3 carrier particle size.

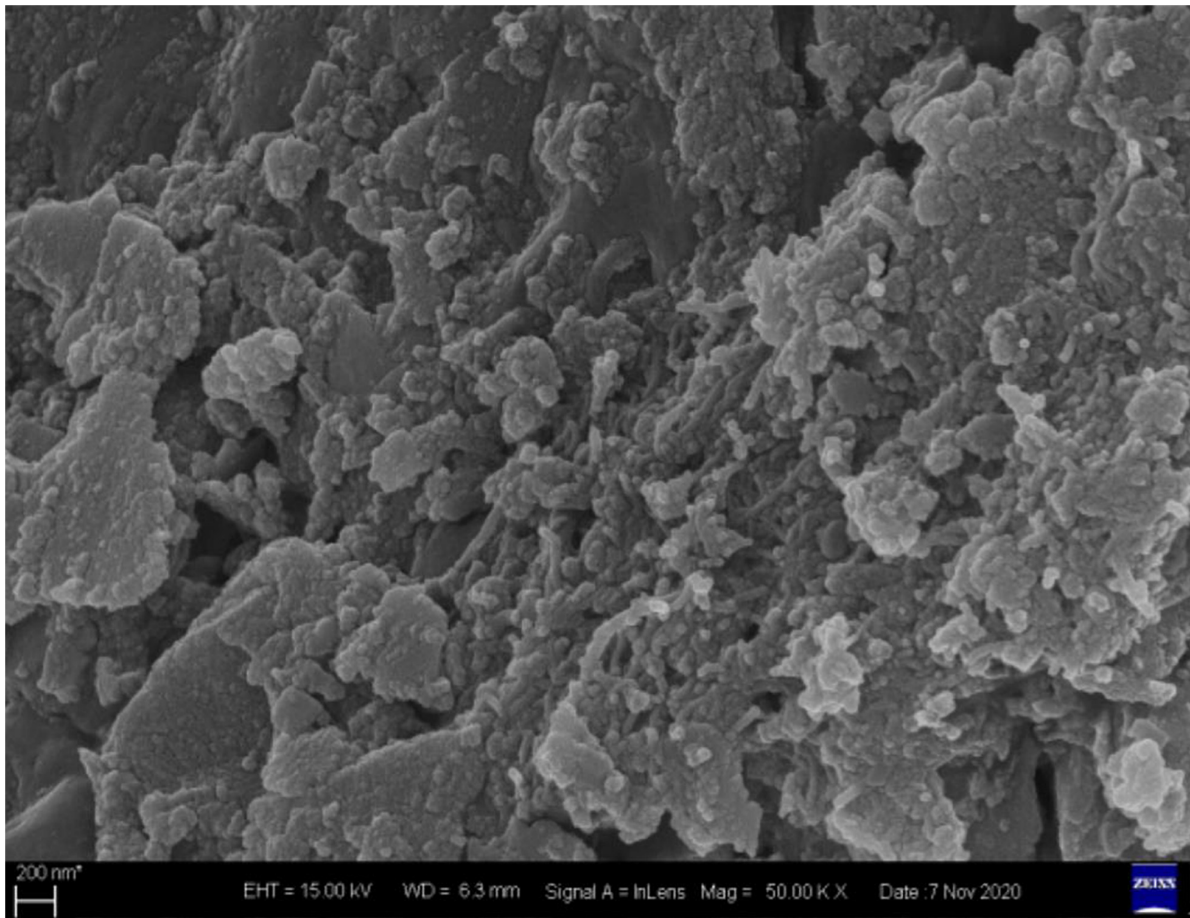


FIGURE 6 | SEM image of the Al₂O₃ carrier-adopted MWCNTs.

supply, and data collector), pure carbon dioxide gas, three-way valve, dynamic test chamber (sensor and sensor base), measuring circuit, interface, and bus. Among them, the dynamic test chamber is the core part of the whole system and the

volume can be adjusted by the external piston (similar to the needle cylinder piston). The target gas concentration is controlled by adjusting the volume of the test chamber and the volume ratio of the injected carbon dioxide gas. The sensor base is fixed on the outside of the test chamber, and the sensor can be quickly switched between the target gas and the air using the piston. This structure solves the disadvantage of slow air exchange in the traditional sensor test chamber and eliminates the influence of gas diffusion on the response time of the sensor.

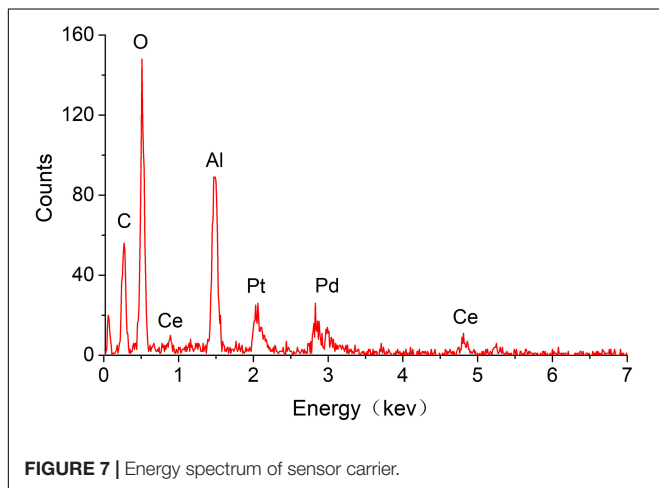


FIGURE 7 | Energy spectrum of sensor carrier.

TABLE 1 | Mapping characterization results.

Element	Weight percent	Atomic percent
C K	4.14	7.23
O K	43.82	57.38
Al K	44.10	34.25
Ce K	5.14	0.77
Pd L	0.80	0.16
Pt M	2.00	0.21
Total	100.00	

Sensor module is a typical Wheatstone bridge for converting gas concentration to voltage output, as shown in **Figure 4**. Fixed resistors R_1 and R_2 (200Ω) are connected to the bridge arm at one end, while the sensing element and compensation element (C, D) are connected at the other end. The two arms are connected to each other using 3.0 V (V_{in}) constant voltage power supply. The detection circuit can output values in millivolts according to the change of target gas concentration. R_3 is a sliding rheostat used to adjust the sensor's zero output value, which is 2000Ω .

RESULTS AND DISCUSSION

Microscopic Characterization of Carbon Nanotubes Modified Nano- Al_2O_3 Carrier

Zeiss Supra 55 scanning electron microscope was used to observe and measure the morphology of multi-walled carbon nanotubes (MWCNTs) and its composite carrier materials with the working voltage of 15,000 V. The micro size of the prepared Al_2O_3 carrier is 20–50 nm, as shown in **Figure 5**. Carbon nanotubes

in Al_2O_3 carrier are intertwined to form a channel inside the carrier, as shown in **Figure 6**. The formation of carbon nanotubes is mainly the mixture of sp^2 -hybridized and sp^3 -hybridized nanotubes. By the influence of quantum physics, it may produce special electrical properties depending on the network structure and diameter. At the same time, carbon nanotubes are also synthesized into carbon-carbon double bonds, hollow cages and closed topologies, so they have excellent thermal and mechanical properties (Dongmei et al., 2014).

Figure 7 indicates that the carrier mainly consists of carbon nanotubes, Al_2O_3 , and CeO_2 . The expected components of carbon nanotubes and CeO_2 have been modified. The mass ratio of Pt and Pd is 2:1 as shown in the mapping characterization results (**Table 1**), while carbon nanotubes reached 4.14%.

Sensor Performance Test

Working Voltage Characteristics of Sensor

Figure 8 shows the sensitivities at different working voltages from 1.5 to 3.0 V with 10% CO_2 . Obviously, the relationship between sensitivity and the working voltage is not linear. With

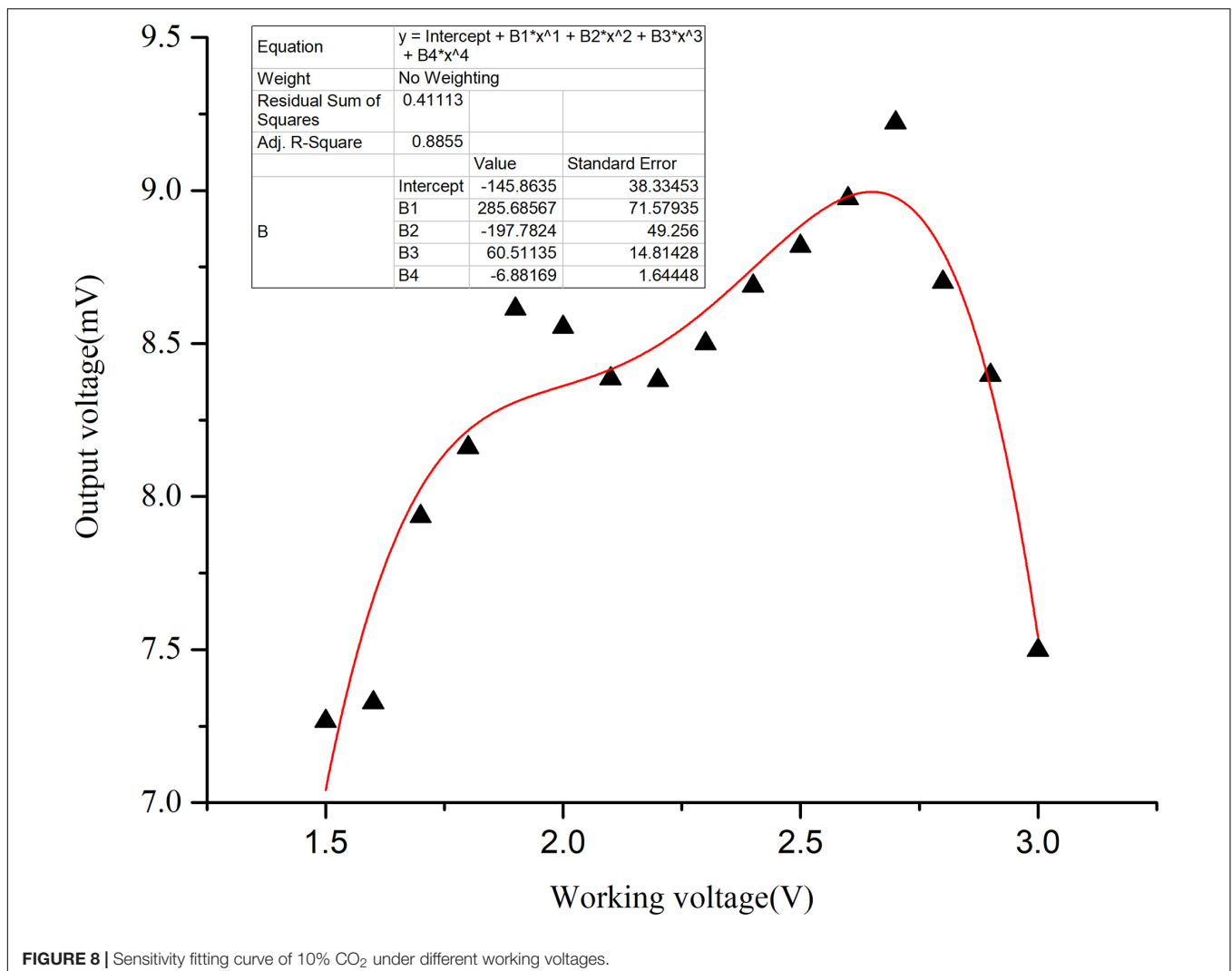


FIGURE 8 | Sensitivity fitting curve of 10% CO_2 under different working voltages.

increased working voltage, the sensitivity first presents a rising tendency and then declines from the fitting sensitivity. The sensitivity reaches its maximum at 2.7 V and thus, the working voltage of 2.7 V is used in subsequent tests. It shows that the MWCNTs-modified sensor can work at a lower voltage and has lower power consumption than the commercial sensor operating at 3.0 V voltage.

Linear Characteristics of Sensor Response

The response recovery curve of CO₂ concentration is determined from 0 to 100% by controlling the CO₂ concentration in the test system at ambient temperature of 25°C and humidity of 45% with an interval concentration of 10% and an operating voltage of 2.7 V by three times. Linear fitting of the average output voltage of the sensor and the CO₂ concentration is shown in **Figure 9** which reveals that the sensor has a good linear relationship ($y = 0.464 + 0.754X$, $R^2 = 0.9986$) with an average sensitivity of about 0.00754 V/10% CO₂. The sensitivity of CO₂ with less than 40% concentration is slightly higher than that with more than 40%.

Consistency Characteristics of Sensor Response

One of the challenges in the application of thermal conductivity gas sensors is the consistency of the response of target gases

with different concentrations, which is affected by both the test conditions and the performance of the sensor. In this test, a dynamic gas chamber with volume scale is used to realize the fast switch between the air and the gas to be measured. The ideal result is then obtained. **Figure 10** shows that the sensor has a good response and recovery performance for different CO₂ concentrations with the same response and recovery characteristics at ten different concentrations. The test results of three times response & recovery time are shown in **Figure 11**, the T₉₀ response time of the developed sensor is 9 s and the T₉₀ recovery time is 13 s.

The Effect of Doping Carbon Nanotubes on Response Characteristics of the Sensor

Response time is an important index of gas sensor. Generally, the time taken for the output value of the gas sensor to reach 90% of the stable value after contacting the target gas is defined as the sensor response time, which is expressed by T₉₀. The smaller the value is, the faster the response speed is. The response time of the sensor is divided into the diffusion of CO₂ gas to the powder metallurgy shell and the direct response time of the sensor to CO₂ gas. The gas diffusion time will be affected by the size of this powder metallurgy overlay. It is determined that 400 mesh specification is the best choice for gas test.

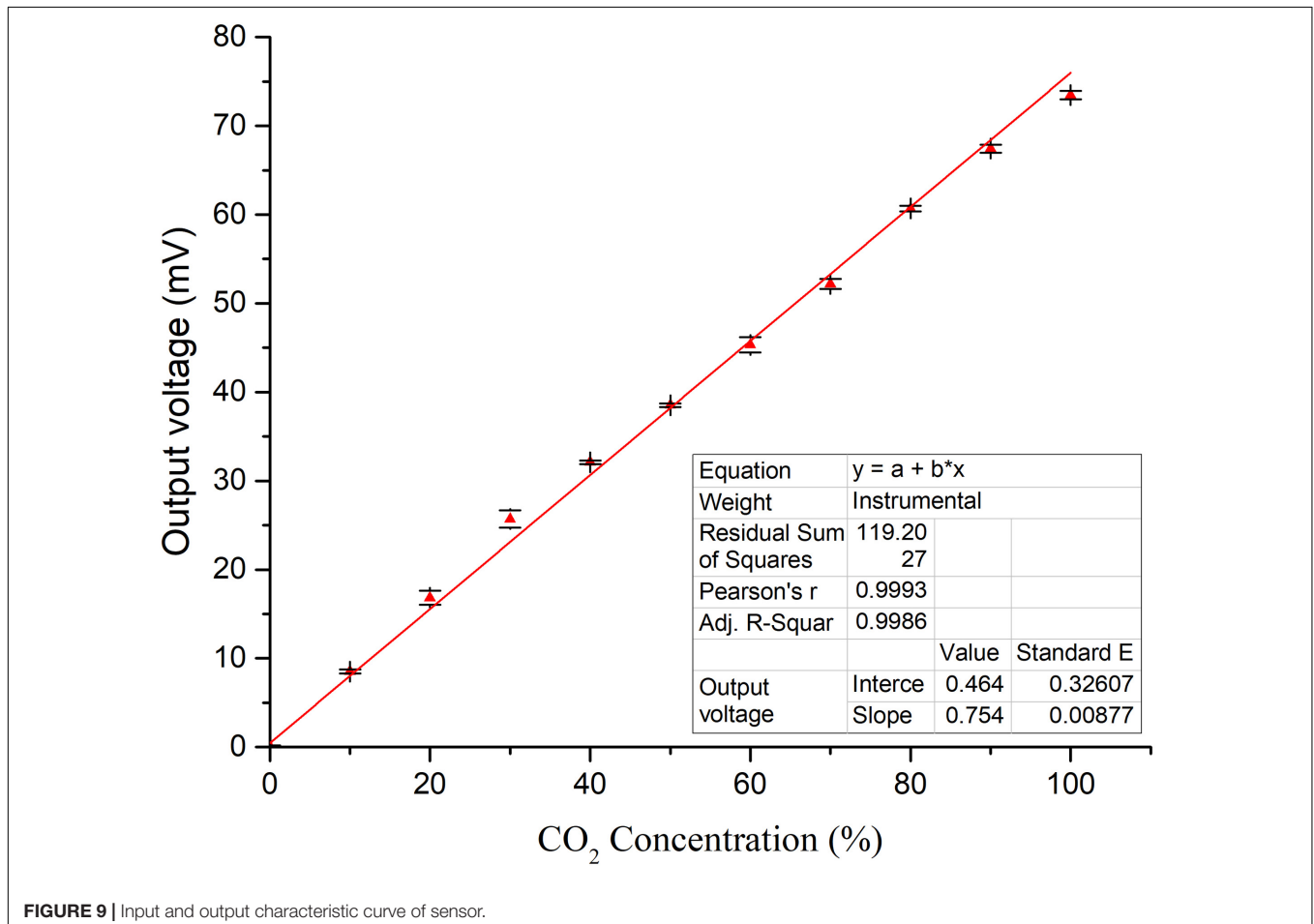


FIGURE 9 | Input and output characteristic curve of sensor.

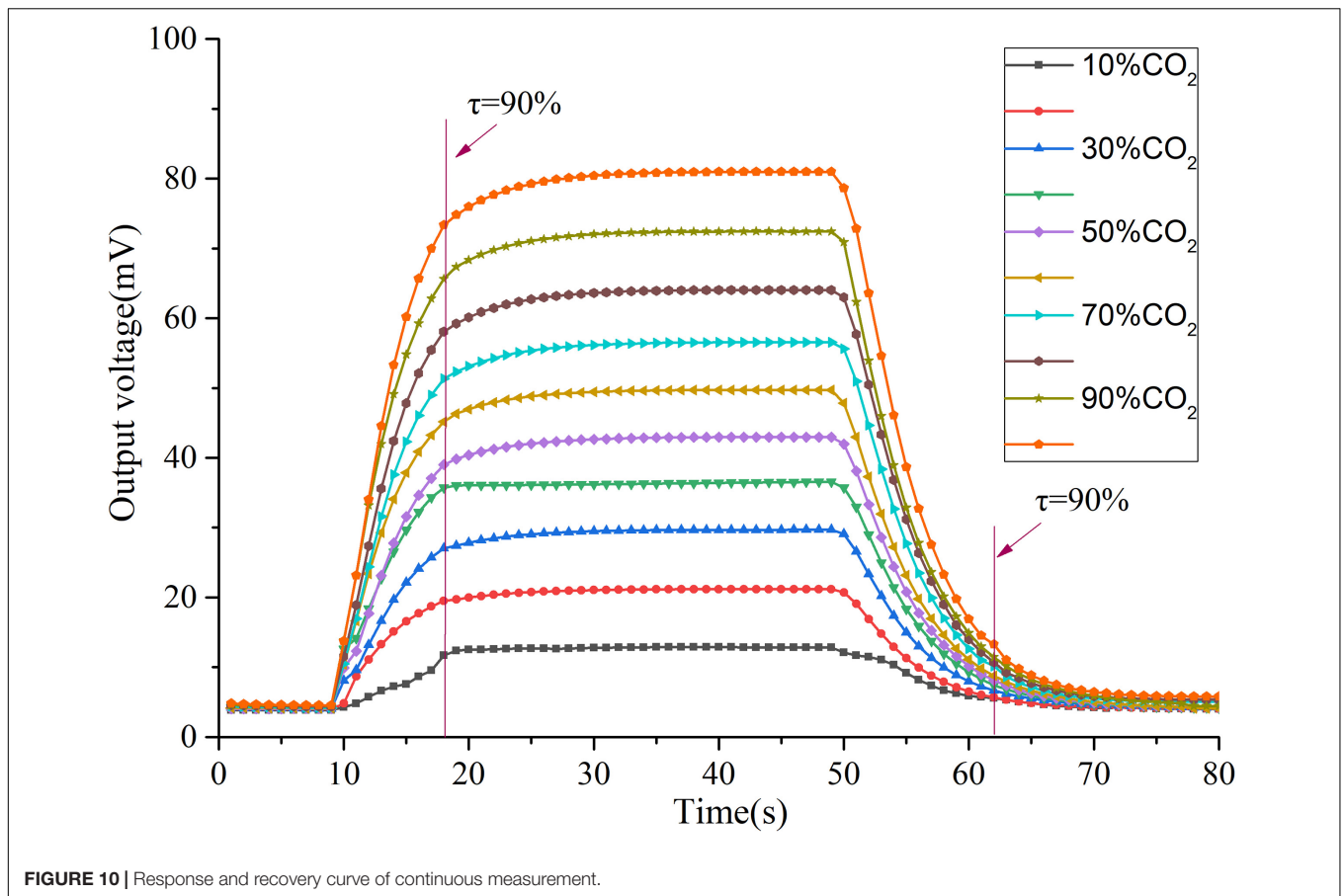


FIGURE 10 | Response and recovery curve of continuous measurement.

Under the condition of 30% CO₂ concentration, the working voltage of the sensor was set as 2.7 V, and the response and recovery curves of three kinds of thermal conductivity sensor

of non-MWCNTs modified, MWCNTs modified and MD62 which produced by Winsen (China) company (Winsen, 2018) with sensitivity of 0.0061 V/10% CO₂ were tested, as shown in Figure 12. The sensitivity of the modified MWCNTs is much higher compared with the sensor before modification and MD62. The T₉₀ response time of the non-MWCNTs modified sensor is 11 s and the T₉₀ recovery time is 17 s. The T₉₀ response time of the MD62 sensor is 10 s and the T₉₀ recovery time is 18 s. The results show that the developed sensor with T₉₀ response time of 9 s and T₉₀ recovery time of 13 s has better performance. Moreover, the modified carbon nanotubes can greatly improve the thermal conductivity sensor's response recovery performance.

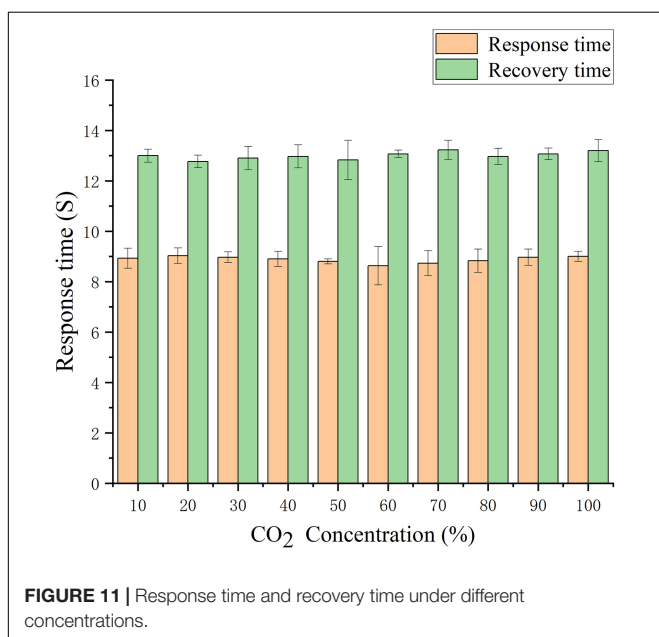
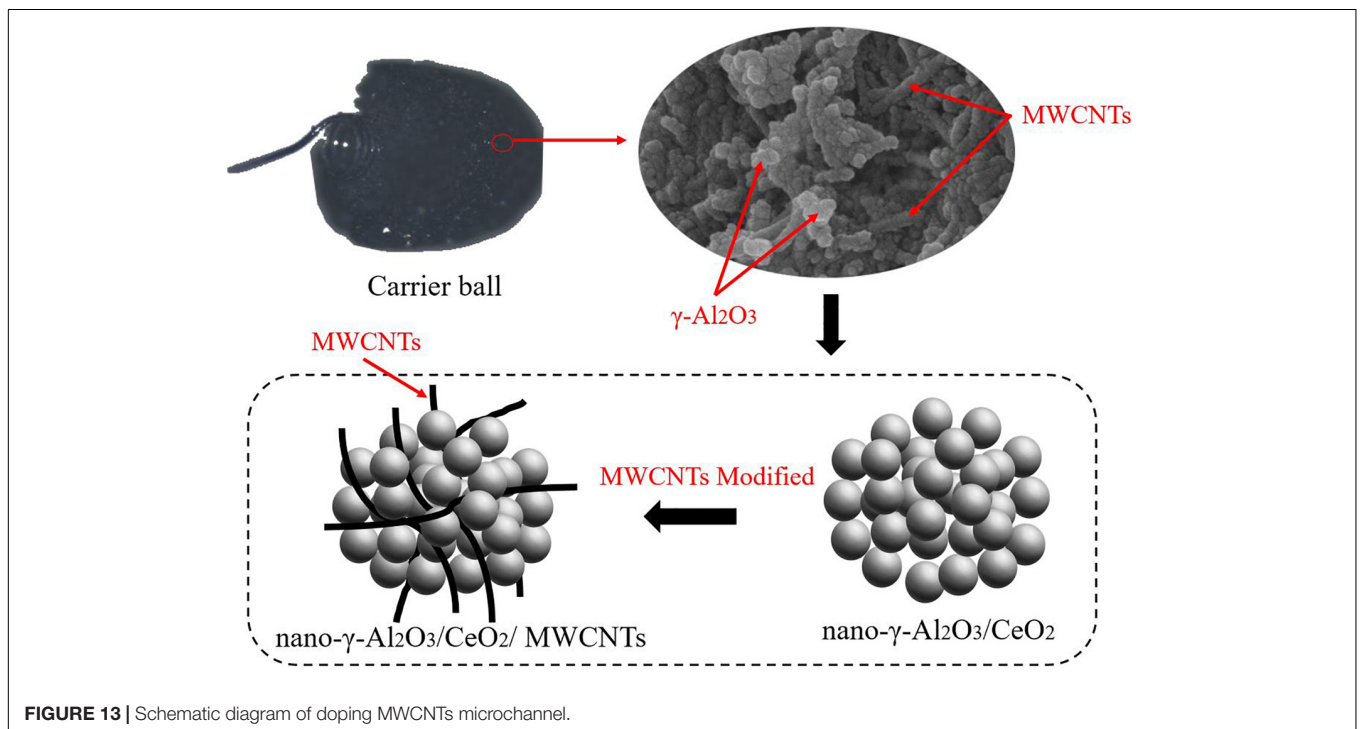
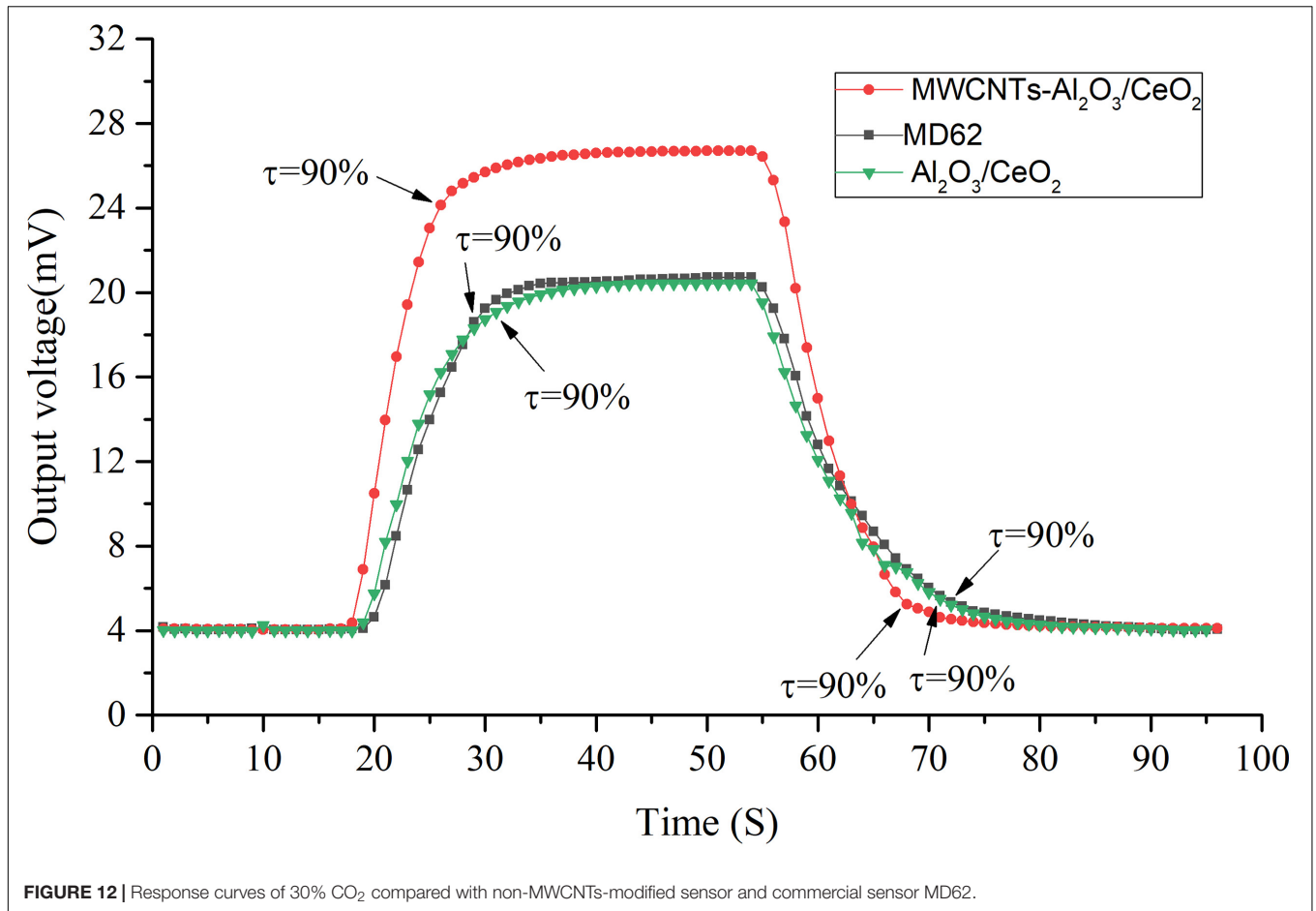


FIGURE 11 | Response time and recovery time under different concentrations.

Fast Response Mechanism Discussion of Thermal Conductivity Sensor

When the target ambient gas changes, making the sensor measurement components quickly achieve a new balance is the key to improve the performance of the thermal conductivity gas sensor. From the response mechanism of the thermal conductivity sensor, the thermal balance ability of the sensing element is the key to affect the response of the sensor, which depends on the thermal conductivity and microstructure of the sensor itself. The traditional way is to reduce the size of the alumina carrier and increase the specific surface area. When the alumina powder is reduced to the nano size (< 50 nm) due to



the carrier formation mechanism, a large number of holes are observed in the inner of the alumina carrier. However, a large proportion of these holes are blind holes and the impermeability of which will lead to gas transmission barrier, resulting in insufficient air exchange and consequently slows down the sensor response speed and easily causes performance drift.

The electrical properties of the sensor carrier can be enhanced through the improvement of carbon nanotubes. First, carbon nanotubes improve the transport efficiency of the measured gas by improving the microchannel of the carrier, as shown in **Figure 13**. The inner diameter of MWCNTs is 2–5 nm, which allows CO₂ gas to diffuse and transport in MWCNTs. MWCNTs and alumina powder are cemented together, which play a role of secondary pore formation and provide a variety of channels for the carrier, so that the original surface expansion heat exchange can be extended to the internal exchange. Second, the current length of carbon nanotubes is 5–30 μm and its thermal conductivity can reach up to 1,000 W/m·K, which is 100 times that of Al₂O₃, thus effectively improving the thermal conductivity of the composite carrier material (Shanni et al., 2020). In addition, because of the large area of carbon nanotubes, it can also accelerate the heat exchange efficiency of the measured gas on the sensitive carrier, greatly improving the response time and stability of the sensor.

CONCLUSION

- (1) The γ -Al₂O₃/CeO₂ carrier material modified by carbon nanotubes is crucial to the sensitivity of the thermal conductivity sensor. Heat treatment can change its crystal form, make it have greater activity and maintain good physical and chemical properties, inhibit carrier agglomeration, and improve the stability of the sensor. Under air conditions, the CO₂ test showed that the average sensitivity of the sensor was 0.0075 V/10% CO₂ and exhibited good linearity.
- (2) For different concentrations of CO₂, the sensor exhibited good response performance and recovery performance, which are similar at different concentrations. The T₉₀ response time and T₉₀ recovery time of the sensor are 9 s and 13 s, respectively, which are better compared to non-MWCNTs-improved sensor and the same type of commercial sensor.

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- (3) Multi-walled carbon nanotubes exhibited thermal conductivity. The modified composite carrier is conducive to the full heat transfer of the measured gas in the hole and improves the heat transfer efficiency of the measured gas on the carrier, which can greatly improve the thermal conductivity of the carrier and enable the sensor to exhibit fast response characteristics.

DATA AVAILABILITY STATEMENT

The original contributions presented in the study are included in the article/supplementary material, further inquiries can be directed to the corresponding author.

AUTHOR CONTRIBUTIONS

BS: conceptualization, investigation, writing–review, project administration, and funding acquisition. XWL: methodology and supervision. FZ and XLL: software. BS and LJ: validation. XQ: resources. FZ and XS: data curation. BS and XLL: writing–original draft preparation and editing. All authors contributed to the article and approved the submitted version.

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