

Design, Fabrication, And Performance Analysis Of VA-SWCNT–ZNO Hybrid Nano Structure-Based Gas Sensors For Low-Temperature Toxic Gas Detection

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Abstract:

This paper presents the design, fabrication, and comprehensive performance analysis of vertically aligned single-walled carbon nanotube (VA-SWCNT)–ZnO hybrid nanostructure-based gas sensors for the detection of toxic gases at low operating temperatures. VA-SWCNTs were successfully synthesized on silicon substrates using the plasma-enhanced chemical vapour deposition (PECVD) technique, followed by uniform deposition of Fe catalyst through RF sputtering. ZnO nanoparticles were subsequently decorated on the CNT surface using the RTCVD method to form hybrid nanostructures with varying weight percentages (0.0 wt%, 2.0 wt%, 6.0 wt%, and 8.0 wt%). The structural, morphological, and quality analysis of the synthesized nanomaterials was carried out using FESEM, HRTEM, and Raman spectroscopy, confirming the formation of well-aligned CNTs and effective ZnO nanoparticle incorporation. The fabricated chemi resistive sensors exhibited excellent gas sensing characteristics towards reducing gas (NH₃) and oxidizing gas (NO₂). A maximum sensor response of 547% was achieved for NH₃ at 30 ppm for the 8.0 wt% ZnO-loaded sensor, along with rapid response and recovery times of 6–8 s and 10–12 s, respectively.

Keywords – Vertically Aligned SWCNTs (VA-SWCNTs), ZnO Nanoparticles, Hybrid Nanostructures, Gas Sensors 'Chemiresistive Sensing, Ammonia (NH₃) Detection, Nitrogen Dioxide (NO₂), Detection 'PECVD Technique, RF Sputtering 'RTCVD 'Sensor Response Response and Recovery Time, Nanomaterials, Low-Temperature Sensing' Surface Functionalization

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I. Introduction & Literature Review

Gas sensors are highly desirable by the scientific fraternity to monitor the safety of the environment from the toxic effects of various hazardous gases like NH₃, CH₄, H₂ etc. which are being released from the various industries and medical sectors. Some toxic pollutants are fatal even at trace level concentration. Various metal oxide semiconductor sensors like TiO₂, SnO₂, ZnO etc. were used to detect these pollutants. However, these metal oxide semiconductor sensors face challenges in the sensor technology. They are being operated at high temperature, though various techniques are being used to get highly sensitive and selective gas sensors at room temperature.

Due to excellent properties of CNTs, they are demanding candidates for gas sensors and field emitters. CNTs possess excellent electrical, mechanical and thermal properties with high aspect ratio and are prominent competitor in the field of nanotechnology. On the basis of their variety of geometry, various applications can be explored in future also. They are finding potential applications in chemiresistive sensors, field emission devices, solar cells, lithium-ion batteries and super capacitors etc. On optimizing the geometrical structure, VA-SWCNTs are considered as the excellent material for field emitter device applications. The properties of CNTs can be enhanced by decorating their surface through various nanoparticles and by making CNT based hybrid nanostructures. Different scientific groups are working on CNT based hybrid nanostructures for high performance gas sensors and field emitters. CNTs can be decorated with Au, Ag, ZnO nanoparticles etc. [to overcome the challenges faced by metal oxide sensors.

Table. 1. Comparative study with Literature

Sensor Material	ppm	Gas	Operating Temp.	Response time	Recovery time	Response	Ref.
ZnO-CNT networks	50 ppm	NH ₃	Room pt.	(30-50 Sec.)	(50-100 Sec.)	425	52
(CNTs-ZnO)	500 ppm	Acetone	Room temperature	(40-50 Sec.)	(50-60 Sec.)	150	53

Ag- -SWCNTs	60 ppm		300C	15 Sec.	68 Sec.	31	11
Au-SWCNTs	40 ppm	NH3	400C	(10-12 Sec.)	(15-20 Sec.)	200	16
Pd-loaded ZnO nanoparticles	40 ppm	NH3	350 °C	(30-40 Sec.)	(95-100 Sec.)	40	51
resent work (VA-SWCNT-ZnO)	30 ppm	NH3	250C	(8-10 Sec.)	(10-12 Sec)	547

II. Proposed Techniques With Methodology

Fe Catalyst Deposition by RF-Sputtering Technique

Si substrates (N-type<100>) were made and ultrasonically cleaned with acetone for 40 min at room temperature and dried. These Si substrates were loaded in the chamber of RF-Sputtering for the uniform deposition of Fe catalyst by keeping the target and substrate distance 10 cm in high vacuum of the order of 10-6 torr and Argon (Ar) plasma was generated at a power rate of 100 W for about 3 min as shown in fig..1.

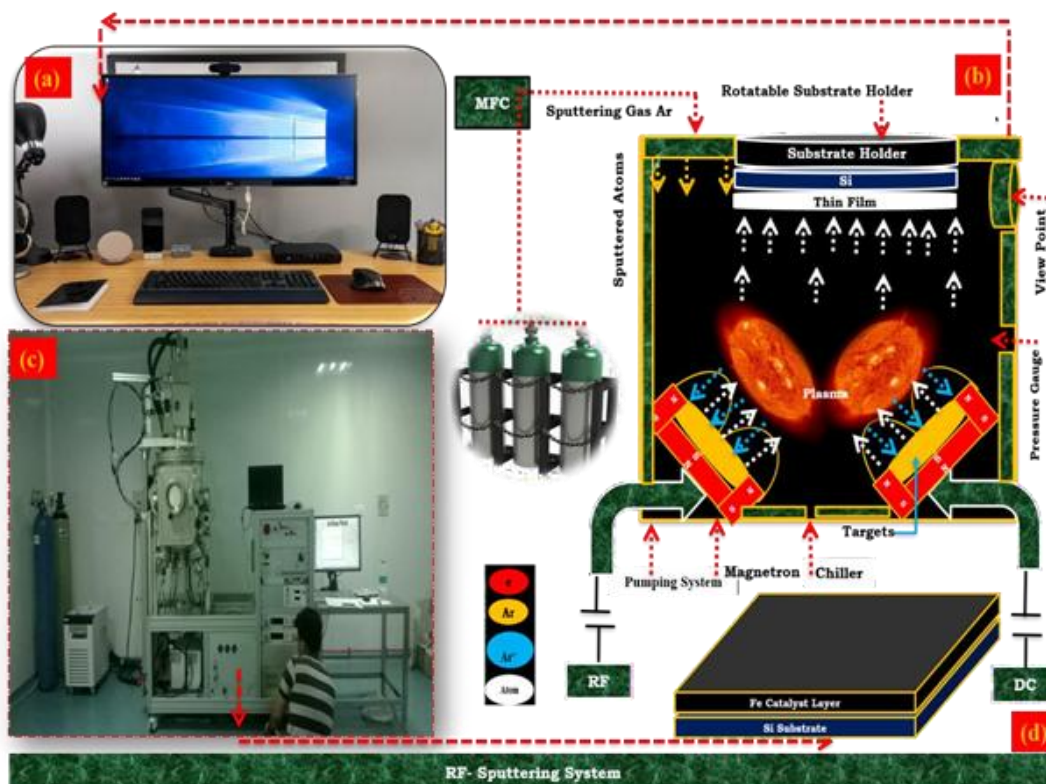


Fig.1. (a) Software attached with RF-Sputtering unit (b) Mechanism of RF-Sputtering (c) RF-Sputtering unit (d) Fe catalyst deposited layer on Si substrate.

VA-SWCNTs Growth by PECVD Technique

The as Fe catalyst-based Si substrate were loaded in the bell jar of PECVD for the growth of VA-SWCNTs under the vacuum of the order of 10-3 torr in the chamber and was pretreated with hydrogen at a temperature of 5000C for 10 min which leads the catalyst break down and the creation of nano islands on Si substrate for the nucleation of carbon atoms. After the pretreatment, the temperature was raised 6500C for the growth of VA-SWCNTs. The acetylene C2H2 was inserted with the flow rate of 20 sccm through the shower head in the bell jar which was associated with NH3 for the dilution and hydrogen was used as carrier gas. The flow rate of hydrogen was maintained about 500 sccm. The unit used with growth mechanism of VA-SWCNTs is shown in fig.. 2.

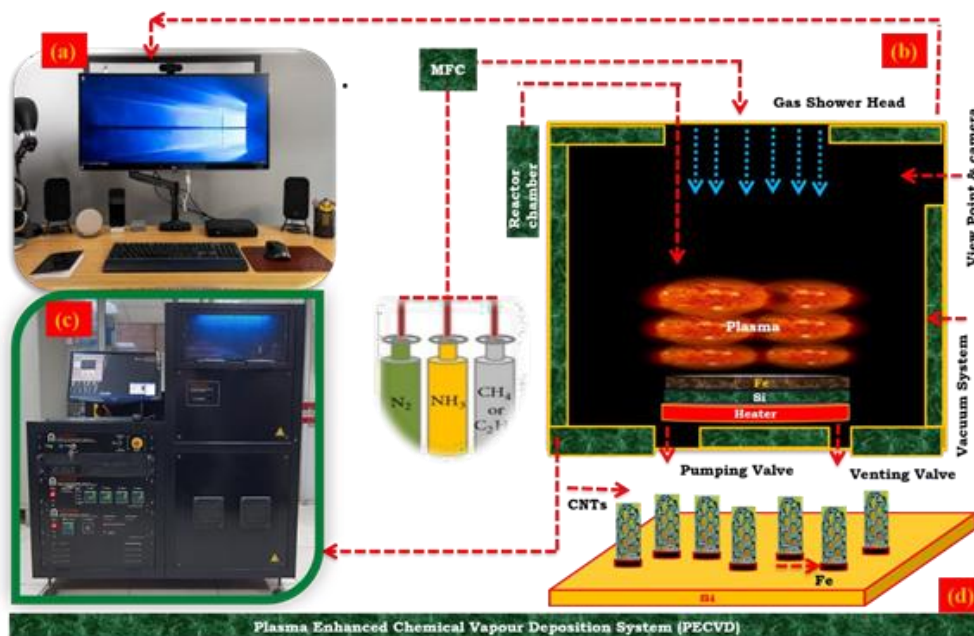


Fig.2. (a) Software attached with PECVD unit (b) Mechanism of PECVD (c) PECVD unit used (d) Grown VA-SWCNTs.

The C₂H₂ gas dissociate into its components with plasma energy and the carbon atoms gets nucleated through Fe nano islands which leads VA-SWCNTs growth. The growth time was about 10 min. The system was cool down and the grown VA-SWCNTs were carried out for further analysis.

Fabrication of VA-SWCNT-ZnO Hybrid Nanostructures

From the as grown VA-SWCNTs, VA-SWCNT-ZnO hybrid nanostructures were grown through RTCVD at an operating pressure of 10⁻⁶ torr. The Zn material was evaporated from the boat through resistive heating in the presence of oxygen (O). The evaporated ZnO material gets attached on the walls of VA-SWCNTs and hence VA-SWCNT-ZnO hybrid nanostructures were obtained as shown in fig .3. The varying wt% of ZnO were 0.0 wt%, 2.0 wt%, 6.0 wt% and 8.0 wt%.

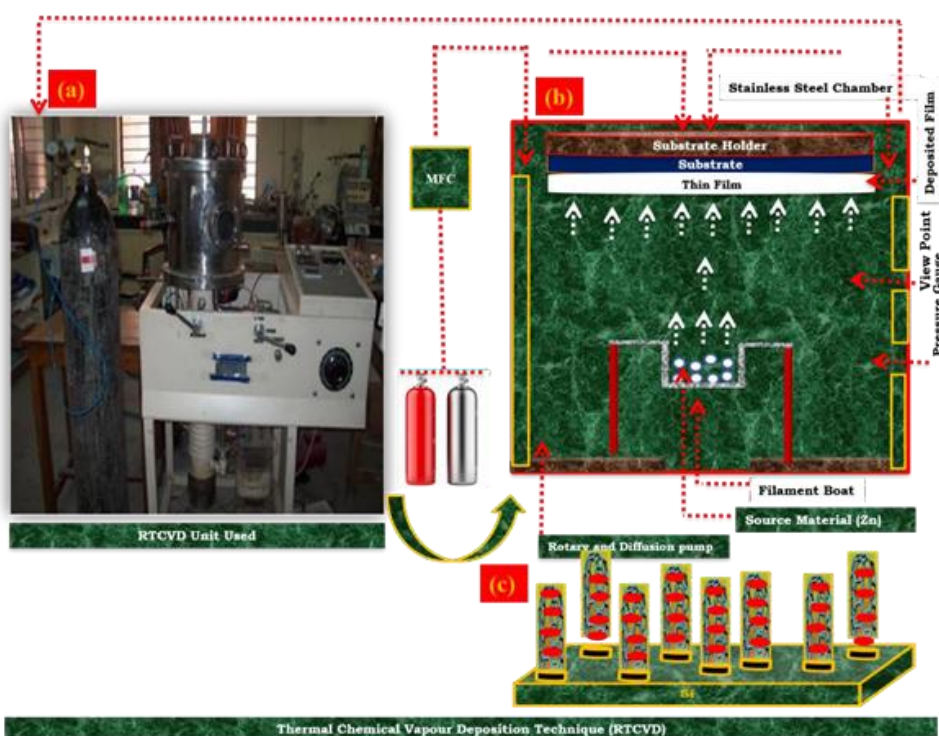


Fig..3. (a) RTCVD unit used (b) Mechanism of RTCVD (c) VA-SWCNT-ZnO structure.

Growth of VA-SWCNTs by PECVD Technique (Flow Chat)

Flow chart for the growth of VA-SWCNT-ZnO nano hybrid structures is shown in fig.4 (a) (b) (c) (d) (e). The importance and explanation of each step of this flow chart has been explained in coming sections.

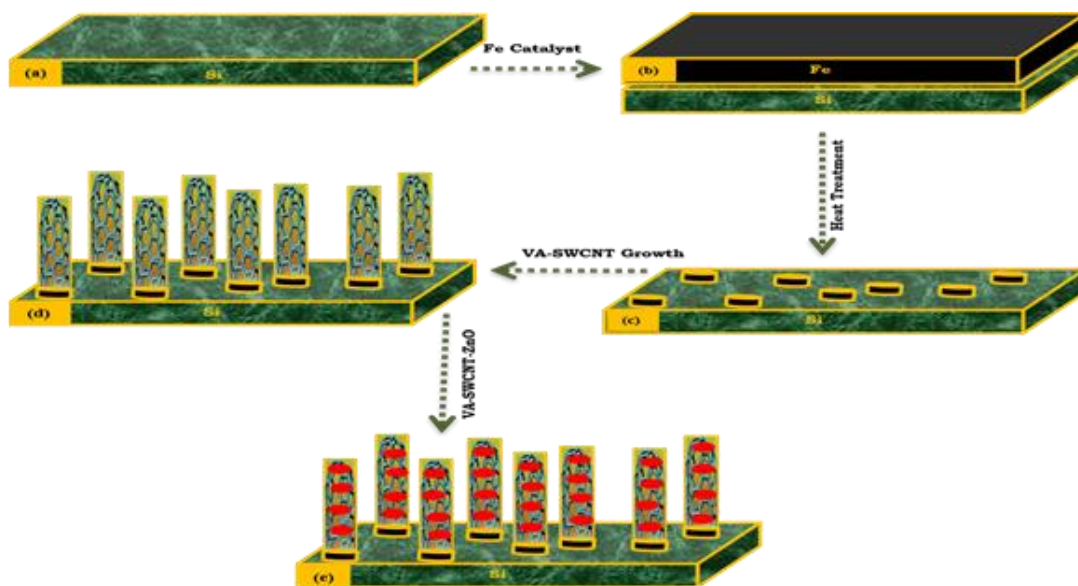


Fig.4. Flow chart for growth mechanism of VA-SWCNT-ZnO nano hybrid structures.

Fabrication of Gas Sensor and Mechanism of Gas Sensing

VA-SWCNT-ZnO Nano hybrid sensor fabrication has been done with the help of gold electrodes having 2 mm gap. The designed VA-SWCNT and VA-SWCNT-ZnO chemiresistive sensor modal and mechanism is shown in fig.5 (a-d).

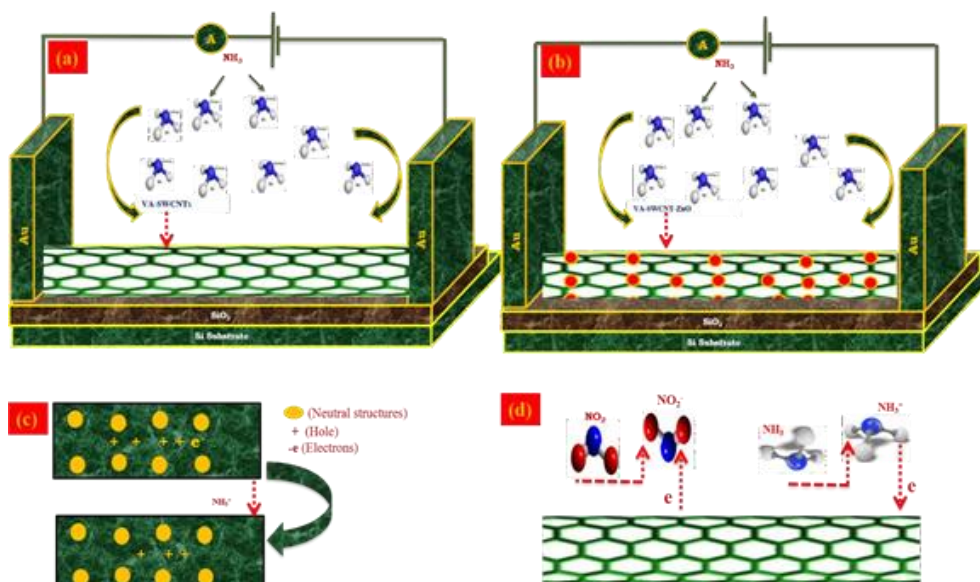


Fig.5. (a-d) Design of VA-SWCNT-ZnO chemiresistive sensor and Mechanism of NH3 and NO2 gas sensing.

Fig...5 represents how electrons are being donated and accepted by reducing and oxidizing gases which leads to band gap variation and the change in resistance occur accordingly.

Gas Sensing Assembly configuration

The self-designed gas sensing assembly interconnected with Keithley Data Acquisition Module KUSB-3100 and a computer was used as shown in fig..6 (a) (b) (c).

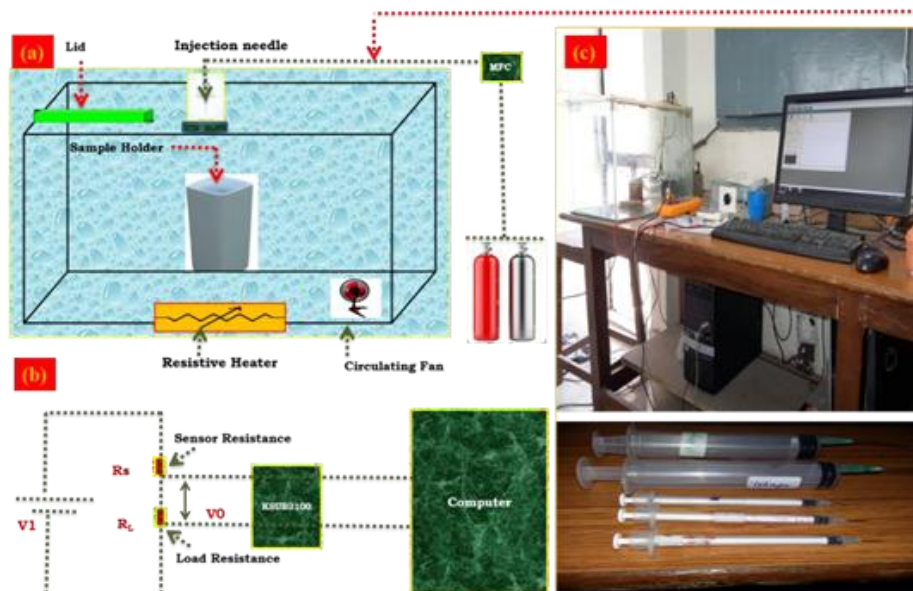


Fig..6. (a) Schematic of sensing assembly (b) Data acquisition system (c) Gas sensing unit.

III. Results And Discussions With Case Studies

FESEM, HRTEM and RAMAN STUDY

The VA-SWCNT-ZnO hybrid nanostructures were observed through field emission scanning electron microscope. FESEM study revealed that good quality VA-SWCNT-ZnO hybrid nanostructures were observed. However, the ZnO % in micrographs was not uniform as shown in fig.4.7 (a-d). Again, HRTEM micrographs revealed that VA-SWCNT-ZnO nanostructures were grown successfully. The nonuniform distribution of ZnO nanoparticles are clearly visible in the micrographs as shown in fig.7 (e-h). The Raman analysis of VA-SWCNT-ZnO with ZnO 0.0 wt% and 2.0 wt% was performed and presented in fig.7 (i) which confirmed the presence of RBM at 196 and 264 cm⁻¹ respectively indicating the successful growth of SWCNTs. The obtained diameter values of these two sample sets using the correlation $d = 248/\nu$ were 1.26 nm and 1.0 nm respectively. G and D-bands were obtained at (1665, 1383) and (1625, 1393) for VA-SWCNTs and VA-SWCNT-ZnO hybrid nanostructure respectively. Calculated ID/IG ratio for these samples were 0.82 and 1.06 respectively, which clearly shows more defects have been created on VA-SWCNTs while hybrid nanostructure production.

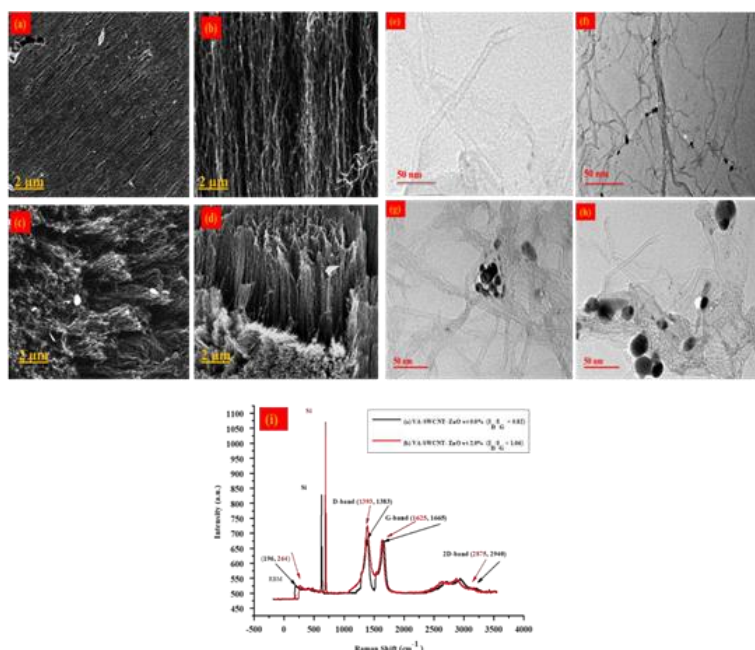


Fig..7. (a-d) FESEM and (e-h) HRTEM micrographs of VA-SWCNT-ZnO with ZnO 0.0 wt% , 2.0 wt%, 6.0 wt%, 8.0 wt% respectively, (i) Raman Spectra of VA-SWCNT-ZnO with ZnO 0.0 wt% and ZnO 2.0 wt%.& Resistance Variation and Sensor Response towards NH3

IV. Conclusion

This work demonstrates the successful design, fabrication, and performance evaluation of VA-SWCNT-ZnO hybrid nanostructure-based chemiresistive gas sensors. The vertically aligned single-walled carbon nanotubes were synthesized using the PECVD technique, followed by Fe catalyst deposition via RF sputtering and ZnO nanoparticle decoration using RTCVD. The hybridization significantly enhanced the sensing characteristics compared to pristine CNTs. Morphological and structural characterization using FESEM, HRTEM, and Raman spectroscopy confirmed the successful growth of high-quality VA-SWCNTs and the effective decoration of ZnO nanoparticles. Raman analysis verified the presence of SWCNTs and indicated an increase in defect density after ZnO incorporation, which contributed to enhanced gas adsorption. The fabricated sensors exhibited excellent sensing performance towards both reducing (NH₃) and oxidizing (NO₂) gases. A maximum sensor response of 547% was achieved for NH₃ at 30 ppm with 8.0 wt% ZnO loading, along with rapid response (6–8 s) and recovery times (10–12 s). The sensors also demonstrated good repeatability, selectivity, and long-term stability.

V. Future Scope

The present study provides a strong foundation for further advancements in CNT-based gas sensing technologies:

- Multi-Gas Detection Systems: Development of integrated sensor arrays capable of detecting multiple gases simultaneously with high selectivity.
- AI-Based Smart Sensors: Integration of machine learning algorithms for intelligent signal processing, pattern recognition, and real-time environmental monitoring.
- Flexible and Wearable Devices: Fabrication of flexible, lightweight, and wearable CNT-based sensors for healthcare and environmental applications.
- Advanced Nanomaterial Hybridization: Exploration of other nanomaterials such as graphene, MoS₂, or conducting polymers with CNTs for improved sensitivity and selectivity.
- Room Temperature Optimization: Further optimization to achieve ultra-high sensitivity at true room temperature without external heating.
- Miniaturization and IoT Integration: Development of compact, low-power sensors integrated with IoT platforms for smart city and industrial safety applications.
- Humidity Compensation Techniques: Designing advanced compensation mechanisms to minimize the effect of humidity on sensor performance.

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