

Novel Gas Ionization Sensors Using Carbon Nanotubes

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Since their discovery in 1991, carbon nanotubes have shown promise for their use as novel micro-nanometre gas ionization sensors. Carbon nanotubes are used as discharge electrodes and can create relatively high electric fields near the nanometer scale tips, lowering the breakdown voltage from several thousand volts to a safe range of several hundreds and even as low as tens volts in comparison to traditional gas ionization sensors. The sensors are sensitive to almost every gas and they can detect various gas species and concentrations. Novel sensors with different anode-cathode separations could potentially be applied to direct identification of gas species in mixtures without the use of mixture separating equipment. But they are limited by nonlinear relations of discharge voltage and current versus gas concentration over wide concentration ranges, resulting in the inability to quantify the gas concentration. The recent research developments on the gas ionization sensors using carbon nanotubes are reviewed in this article, and potential solutions to the problems associated with novel sensors are also suggested.

Keywords: Carbon Nanotube, Gas Sensor, Gas Ionization, Discharge Characteristics.

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1. INTRODUCTION

Gas sensing has a variety of applications such as environmental monitoring, sensing in chemical processing plants, gas detection for counter-terrorism, and sensing in navigation and spaceflight, and therefore it is of significant importance for human health, the safe running of industrial process and the security of society.¹⁻⁷ Gas sensors, such

as metal oxide semiconductor (MOS) sensors,⁸⁻⁹ semiconductor sensors,¹⁰⁻¹⁵ electrochemical sensors,¹⁶⁻²⁶ thermal conductivity detectors (TCD),²⁷⁻²⁸ capacitor based chemical sensors,²⁹⁻³¹ carbon nanotube (CNT) based chemical sensors,³²⁻³⁵ optical sensors,³⁶⁻⁴⁹ flame ionization sensors,⁵⁰⁻⁵² photoionization detectors⁵³⁻⁵⁵ and discharge ionization detectors,⁵⁶⁻⁵⁷ have shown several disadvantages such as low sensitivity, poor selectivity and stability, complex structure, high working voltage and high cost, and therefore consequently can not meet industrial demands.

Gas ionization sensors using carbon nanotubes as a discharge electrode have been developed for gas detection since 2001.⁵⁸ Carbon nanotubes with a tip radius in the nanometer scale, increase the intensity of the electric field in the electrode gap at a relatively low voltage, and thereby lower breakdown voltages of the novel sensors from several thousand volts to a safe range of several hundred volts and even as low as to tens volts,^{1,58-59} when compared with traditional ionization sensors with traditional plate-plate electrodes. CNT sensors with different electrode separations have been fabricated to detect the identity of gas species in mixtures directly without separating mixtures, and sensor with a given separation can be chosen to identify one component gas.⁶⁰

Different gases show different conductance ability in ionization coefficients (the first, second, and third

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Townsend coefficients of ionization) and ionization potentials.⁶¹ As a result, they tend to be ionized at different discharge voltages and currents across the electrode gap.^{1,60} Studies have also shown that the discharge characteristics, which are expressed as the relationship between gas concentration with discharge voltage and discharge current, exhibit linear relationships within small concentration ranges of single gases,^{62–64} but exhibit nonlinear characteristics over wide concentration ranges.^{1,60,78} In addition, high thermal consumption under a large discharge current tends to give rise to significant catastrophic damage to the carbon nanotube film,⁶⁵ leading to negative influence on the stability and life-span of the sensors. Therefore future studies are needed to improve the stability and life-span of the gas ionization sensor using carbon nanotubes (CNTGIS). The CNTGISs in the future are expected to offer several advantages such as linear discharge characteristics, repeatable results, low cost of the sensor system, and ease of use under most conditions.

2. GAS IONIZATION SENSORS USING CNT FILM AS DISCHARGE ELECTRODE

Two reports have been published on the successful design of a novel electronic device, with one report on a novel gas sensor using CNT film as the cathode⁵⁸ in 2001 and the other on a novel gas sensor using CNT film as the anode¹ in 2003. CNT film, used in the novel sensors to take place of one of the traditional plate electrodes, generates very high electric fields and significantly lowers the breakdown voltages from several thousand volts to several hundred volts in comparison to traditional plate electrodes,^{1,58} as shown in Figure 1.¹ Figure 2⁵⁸ also indicates that different gases have different conductance, resulting in different discharge voltages and currents. The above findings have propelled the CNTGIS towards its practical usage in measuring gases and mixtures. Subsequently, many research groups have conducted studies on the structural design and discharge characteristics of the novel CNTGISs.^{59–60, 62–64, 66–78}



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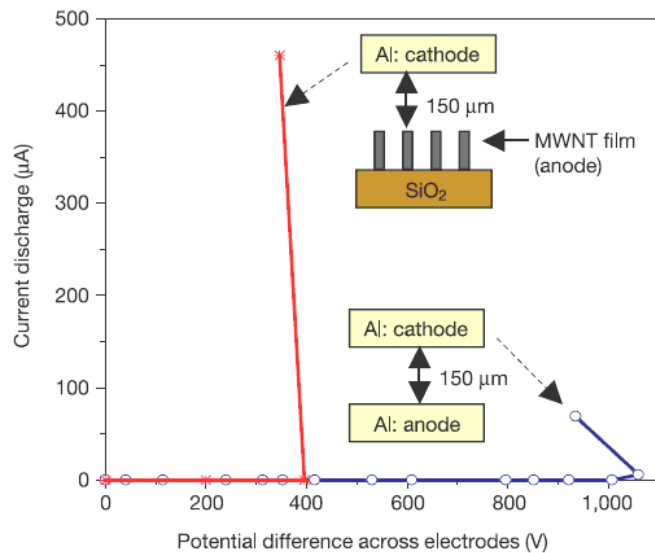


Fig. 1. Comparison of current–voltage (I – V) characteristics for electrical breakdown between the novel sensor using CNT and the traditional sensor. I – V curve (blue) of the traditional sensor with Al plate as the anode, separated from cathode by $150\ \mu\text{m}$ (micrometer), showing breakdown voltage of air at $960\ \text{V}$ with discharge current of $69\ \text{mA}$ (milliampere); of the novel sensor with MWNT film as anode (red) showing breakdown voltage of air at $346\ \text{V}$ with discharge current of $460\ \mu\text{A}$ (microampere). Reproduced with permission from [1], A. Modi et al., *Nature* 24, 171 (2003). © 2003, Nature Publishing Group.

Previous studies have basically displayed the two structures of CNTGIS, one with plate–plate electrodes^{1, 58–60, 69–70} and the other with tip–plate electrodes.^{62, 66–69} In the plate–plate structure shown in Figure 3(a), CNTs are grown on the semiconductor or metal substrates, such as porous silicon,⁵⁸ silicon,^{1, 60, 69, 79} porous alumina,^{62–64} Cr/Au,^{59, 74} Fe/Ti,⁷⁸ and Ta.⁷⁹ In the tip–plate structure shown in Figure 3(b), CNTs are grown on a tungsten tip or stainless-steel tip as one electrode,^{66–68} while the other electrode is a plate. Both structures have exhibited the potential for gas sensing.

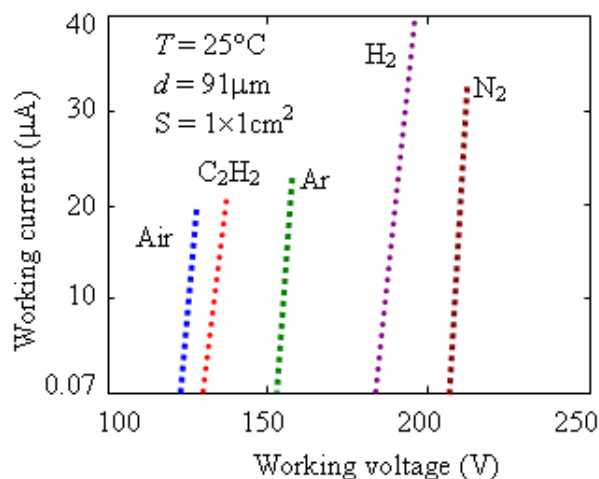


Fig. 2. I – V characteristics of several kinds of single gases. Reproduced with permission from [58], Y. Zhang et al., Study of gas sensor with carbon nanotube film on the substrate of porous silicon, *14th Intl. Vac. Microelectron. Conf.*, University of California, Davis, CA, USA (2001), pp. 13–14. © 2001, IEEE.

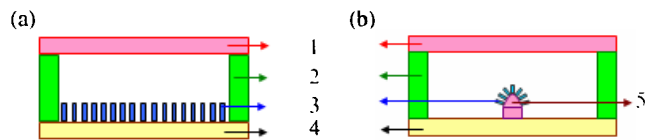


Fig. 3. The electrode structures of the novel gas ionization sensors using CNT. (a) plate–plate electrode, with CNT film grown on a plate substrate as one electrode. (b) Tip–plate electrode, with CNT film grown on a tip as one electrode. (1—Electrode plate 1st, 2—Insulator, 3—CNT, 4—Substrate (Electrode plate 2nd), 5—Tip).

3. EFFECT OF CNT GROWING TECHNIQUES

In general, CNTs are grown by thermal chemical vapor deposition (TCVD) or chemical vapor deposition (CVD). Different deposition techniques result in different

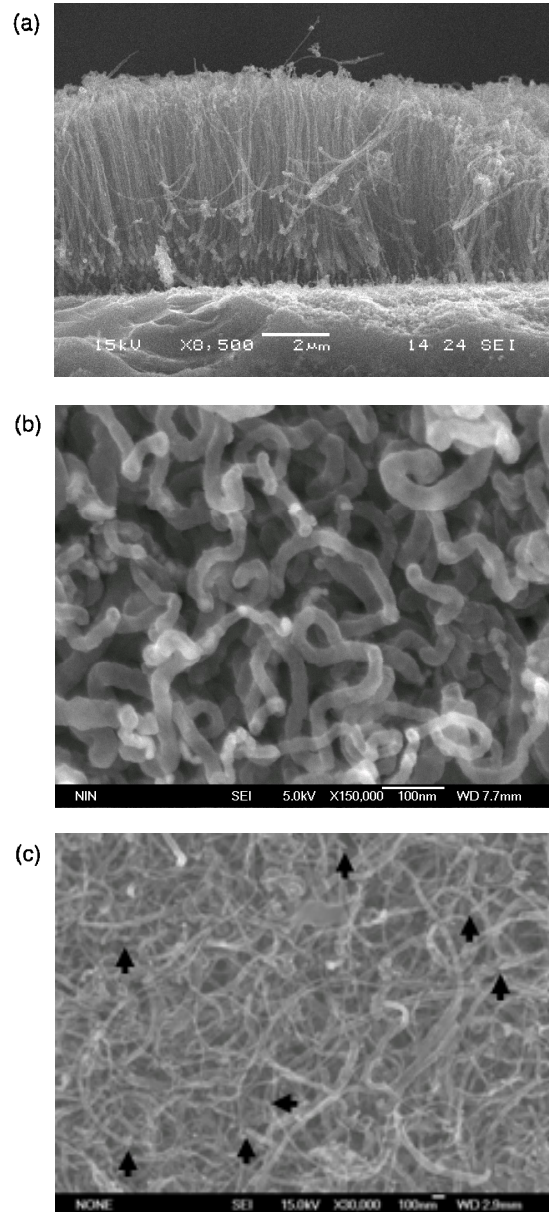


Fig. 4. Different shapes of CNTs fabricated by three different techniques. (a) thermal chemical vapor deposition, TCVD; (b) chemical vapor deposition, CVD and (c) screen printing, SP.

diameters and different growing directions of CNT. TCVD produces CNT with a diameter of more than 50 nm aligning vertically on the substrate,^{60, 79–80} as shown in Figure 4(a). CVD produces CNT with a smaller diameter of 10–100 nm, in continuous cirrus shape and with no tip,⁸¹ as shown in Figure 4(b). Besides TCVD and CVD, screen printing (SP) is also used to fabricate CNT film.^{59, 74} In SP, CNTs grown by CVD are cut short and the slurry of the short-cut CNTs mixed with organic solution is printed on substrates. Thus, SP produces CNTs of the same diameter and similar cirrus shape as produced by CVD, as shown in Figure 4(c), where part of the tips of the short-cut CNTs can be seen along the arrows.

An interesting phenomenon occurs of which that the CNT of a smaller diameter tended to generate higher power discharges, such as corona¹ and arc discharge,⁸² while the CNT of a larger diameter produced lower power discharges,^{58, 60, 69–70, 78} such as pre-discharge before breakdown or self-sustaining dark discharge (viz. Townsend discharge). This phenomenon can be explained by the calculation simulation of the uniformity of the electric field distribution.⁶⁹ The non-uniformity coefficient f is used to express the uniformity of the electric field and calculated by the ratio of maximum electric field intensity to average electric field intensity between two electrodes.^{83, 84} When f is larger than 4, the electric field distribution in the gap is extremely non-uniform, leading to the easy generation of a high power discharge such as corona discharge.⁸³ CNTs with a small diameter, when compared with large diameter ones, tend to result in high non-uniformity coefficients ($f > 4$) owing to the stronger tip effect of CNTs, and thereby the easy formation of higher power discharges. The results indicate that the more non-uniform the distribution of the electric field in the gap is, the more easily a

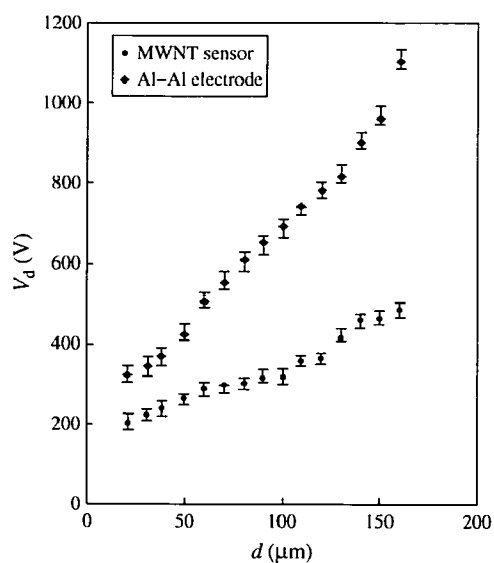


Fig. 5. Effect of interelectrode spacing on breakdown voltage (V_d and d denote breakdown voltage and interelectrode spacing, respectively). Reproduced with permission from [71], G. H. Hui et al., *Meas. Sci. Technol.* 17, 2799 (2006). © 2006, Institute of Physics Publishing Ltd.

high power discharge with high current and large thermal consumption is generated.⁶⁹ But a high discharge current and large thermal consumption at extremely non-uniform electric fields could cause catastrophic damage such as the evaporation or breakage of CNTs,⁶⁵ leading to the stability and life-span of the sensor to be negatively influenced. Therefore the growing technique of CNT is of significant importance for the performance of the CNTGIS.

4. EFFECT OF INTERELECTRODE SPACING

Experimental studies of the effect of anode–cathode separation have shown that the breakdown voltage of the novel sensor increases significantly with electrode separation and is faster than traditional gas ionization sensors with Al–Al plate electrodes,^{60, 71–72} resulting in the sensitivity to electrode separation higher than that of the traditional sensor, as shown in Figure 5.⁷¹

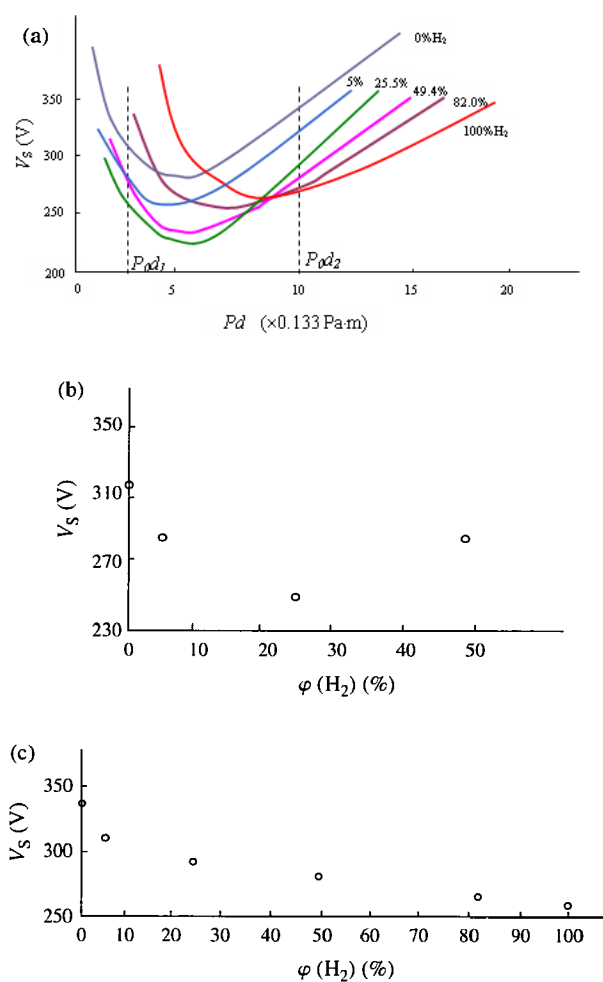


Fig. 6. Effect of electrode separation on the discharge characteristics of H_2 in a mixture with N_2 . (a) V_s – Pd curves (V_s , P and d denote breakdown voltage, gas pressure and electrode separation, respectively). Reproduced with permission from [61], X. J. Xu and D. C. Zhu, *Gas Discharge Physics*, Fudan University Press, Shanghai, China (1996). © 1996, Press of Fudan University of China. V_s – $\varphi(H_2)$ curves (with $\varphi(H_2)$ denoting H_2 concentration) at interelectrode gap of (b) d_1 and (c) d_2 , respectively.

In addition, previous experiments have also shown that the linearity of discharge characteristics of traditional ionization sensors with plate-plate electrodes is affected by electrode separation. For a H_2-N_2 mixture, breakdown voltage V_s as functions of Pd (with P and d denoting gas pressure and electrode separation, respectively) shown in Figure 6(a)⁶¹ indicates that at a controlled pressure P , the breakdown voltage V_s has a nonlinear and linear relation with the H_2 concentration $\varphi(H_2)$ at the interelectrode spacings d_1 and d_2 , respectively, as shown in Figures 6(b) and (c). For an $Ar-N_2$ mixture, the discharge characteristics are also affected by electrode separation.⁶¹ Therefore the interelectrode spacing is of significant importance for breakdown voltage as a linear function of gas concentration, and thus, the effect of electrode separation should not be ignored in experiments.

5. SPECIES AND CONCENTRATION RANGES OF DETECTED GASES

The gases detected by the CNTGISs, excluding Air and CO_2 , are divided into two categories: the inert gases and the flammable, explosive and poisonous gases. More research has been carried out on the former gases than on the latter. Six research groups have studied the discharge characteristics of inert gases such as N_2 , Ar, and He.^{1, 58-60, 66, 69-78, 82} But up to now, only three research groups have studied the discharge characteristics of dangerous gases such as H_2 , C_2H_2 , NH_3 , CH_4 , CO, alcohol, acetic acid, and acetone.^{1, 60, 62, 69, 82} The reason for this is mainly for considerations of safety. The CNTGIS works in discharge states, and high power discharge at extreme non-uniform electric fields may result in fire and explosion of flammable and explosive gases when concentration rises to the fire and explosion limits. The other safety problem lies in the difficulty of complete isolation of poisonous gases from people and appropriate processing of dangerous gases after detection.

The concentration ranges of the previously detected gases and mixtures are classified into wide and narrow ones. Gases over wide concentration ranges include 0–50032 ppm (microlitre/litre) CH_4/N_2 , 0–500 ppm CO/N_2 , 0–350 ppm H_2/Air ,^{58, 60, 69} 10^{-6} – 10^{-1} mol/L Air, Ar, N_2 and NH_3 ,¹ 0–100% Ar/Air ,^{1, 71} and NH_3/Air ,¹ 0–100% N_2/Ar , 0 – 12×10^{-5} mol/L alcohol and acetic acid in dry Air,⁶² 10^{-6} – 10^{-3} mol/L Air and Ar,⁷⁸ 10^{-7} – 10^{-4} mol/L Air⁷⁸, etc., while gases within small concentration ranges include 0–20 ppm C_2H_2/Air ,⁵⁸ 0–40 $\mu g/L$ acetic acid, alcohol, acetone and water vapor in dry air,⁶² etc.

6. QUALITATIVE IDENTIFICATION OF GAS COMPONENTS

Previous experiments have shown that discharge voltage range varies significantly with interelectrode spacing.⁶⁰

Table I. The ranges of discharge voltages of the four sensors with different interelectrode spacings in CH_4-CO mixtures.

Range of discharge voltage (V)			
Range 1		Range 2	
CH ₄ (0–50000 ppm)/ CO(0–500 ppm)		CH ₄ (0–15000 ppm)/ CO(0–50 ppm)	
Sensor 1	Sensor 2	Sensor 3	Sensor 4
59.9–279.6	79.7–339.0	230–300	210–380

Four sensors with different electrode separations have been used to measure the discharge characteristics in CH_4-CO mixtures. The discharge voltage ranges corresponding to the two different concentration ranges shown in Table I are significantly different, suggesting that the CNTGIS array with different electrode separations could potentially be used to detect the identity of every component gas in mixtures. As expected, the two components, CH_4 and CO in the CH_4-CO mixtures within the two concentration ranges in Table I, have been identified qualitatively by using data fusion technology, respectively.⁶⁰

7. QUANTITATIVE DETECTION OF GAS CONCENTRATIONS

Several research groups have studied the discharge characteristics of CNTGISs on the exposure to the above mentioned gases in given concentration ranges. The effects of concentration on discharge voltage and discharge current at breakdown have been studied over wide ranges of 0–50032 ppm CH_4/N_2 and 0–500 ppm CO/N_2 , as shown in Figures 7(a–d).⁶⁰ But the four curves in Figure 7 are all nonlinear, indicating that it is difficult to quantify gas concentration over a wide concentration range.

In contrast to the nonlinear curves over wide concentration ranges,^{1, 60, 71, 78} the curves of breakdown voltage and pre-discharge current (discharge current before breakdown) as functions of gas concentration within small concentration ranges are linear,⁶² as shown in Figures 8(a, b)⁶² and Figure 9.⁶² In comparison to Figure 7, Figure 8 indicates that linear breakdown discharge characteristic only exists within a small concentration range. The results shown in Figure 9 are worthy of further consideration and suggest that the pre-discharge characteristics (discharge characteristics before breakdown) within small concentration ranges could potentially be used as a reference method for the study of discharge characteristics over wide ranges of gas concentrations.

8. REPEATABILITY, STABILITY, SELECTIVITY AND LIFE-SPAN OF THE SENSOR

Experimental studies^{61, 71} have shown that environmental factors, such as trace impurity gases, temperature and

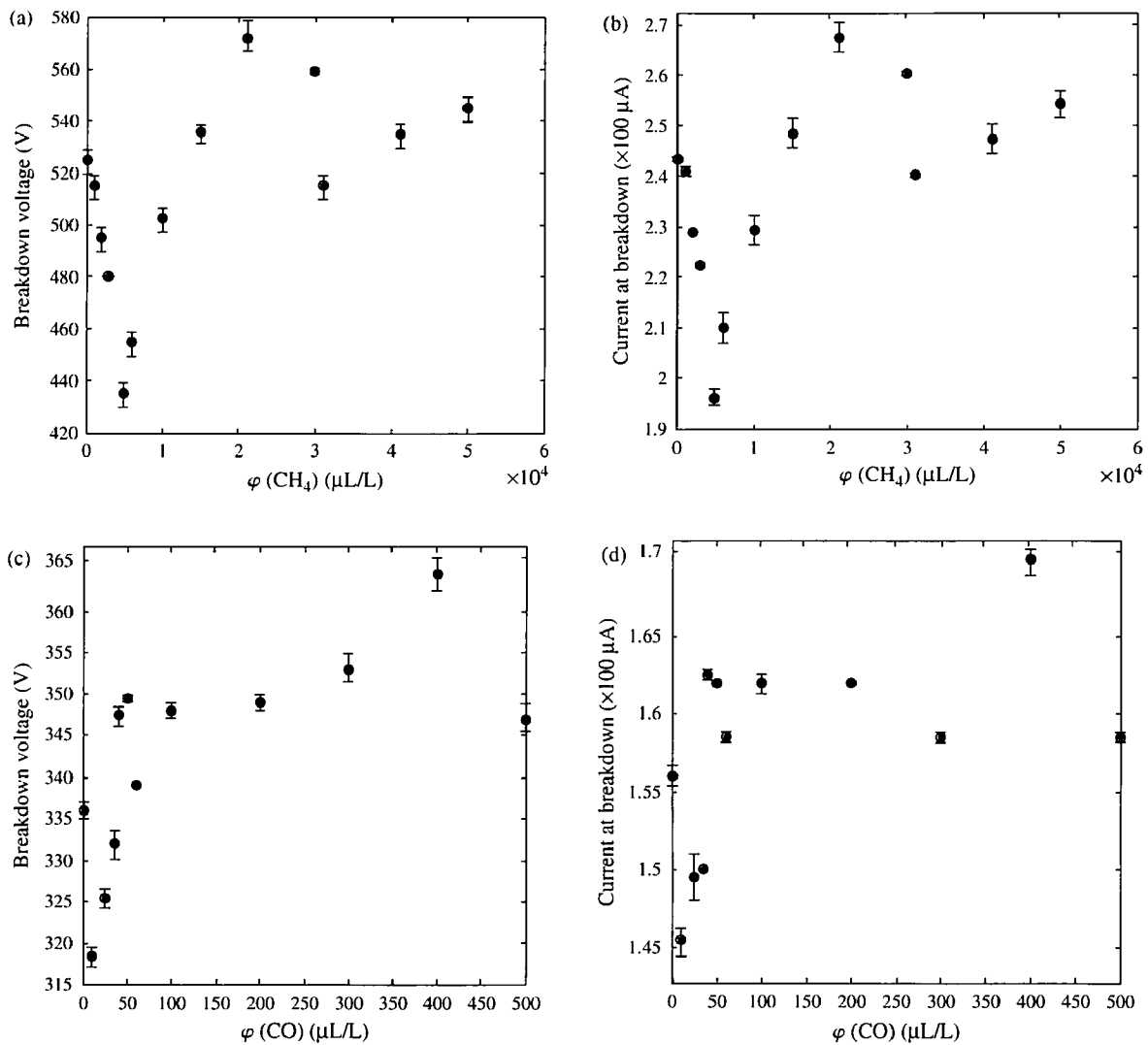


Fig. 7. Effect of gas concentration on discharge characteristics over wide ranges of 0–50032 ppm CH_4/N_2 and 0–500 ppm CO/N_2 . (a) breakdown voltage and (b) discharge current at breakdown as nonlinear functions of CH_4 concentration in a CH_4 – N_2 mixture; (c) breakdown voltage and (d) discharge current at breakdown as nonlinear functions of CO concentration in a CO – N_2 mixture. Reproduced with permission from [60], Y. Zhang et al., *Sens. Actuators, A* 125, 15 (2005). © 2005, Elsevier B. V.

humidity, affect the discharge state of the detected gases and give rise to cross-sensitivity of the CNTGIS. The cross-sensitivity is explained as follows. When temperature and humidity maintain constant, discharge voltages and discharge currents of all sensors (e.g., two sensors on exposure to a CH_4 – CO mixture⁶⁰) vary with concentration of one detected component although concentrations of the other components maintain constant. On the other hand, when concentrations maintain constant, a change in temperature or humidity leads to the change of discharge voltages and discharge currents of all sensors. Trace impurity gases in the detected environment also exhibit the similar effect on discharge voltage and discharge current with temperature or humidity. Therefore the cross-sensitivity of the CNTGIS will cause measurement errors and thereby negatively affect the repeatability, stability and selectivity of the CNTGIS. For example, the gas ionization sensor using CNT film cathode (CNTFCGIS) has been used to detect CH_4

in 0–4937 ppm CH_4/N_2 under constant temperature and humidity, and shows a repeatability error of 11.1% through the calculation analysis of breakdown voltage as a function of concentration, shown in Figure 7(a).⁶⁰ The repeatability error is calculated by a ratio of the maximum deflection (549 ppm) of the measured concentrations to the upper limit (4937 ppm) of the gas concentration range. The sensor also has a repeatability error of 8.4% for the experimental results of the relation between discharge current at breakdown and concentration, shown in Figure 7(b).⁶⁰ Additionally, the CNTFCGIS used in the detection of 10–50 ppm CO/N_2 shows repeatability values of 10.0% and 24.0% for the relations shown in Figures 7(c) and (d), respectively. Such large repeatability errors indicate the negative effect of the environmental factors on the CNTGIS.

Study results have also shown that the increased discharge current resulted in increased thermal consumption of CNTs, easy damage to CNTs⁶⁵ and consequently

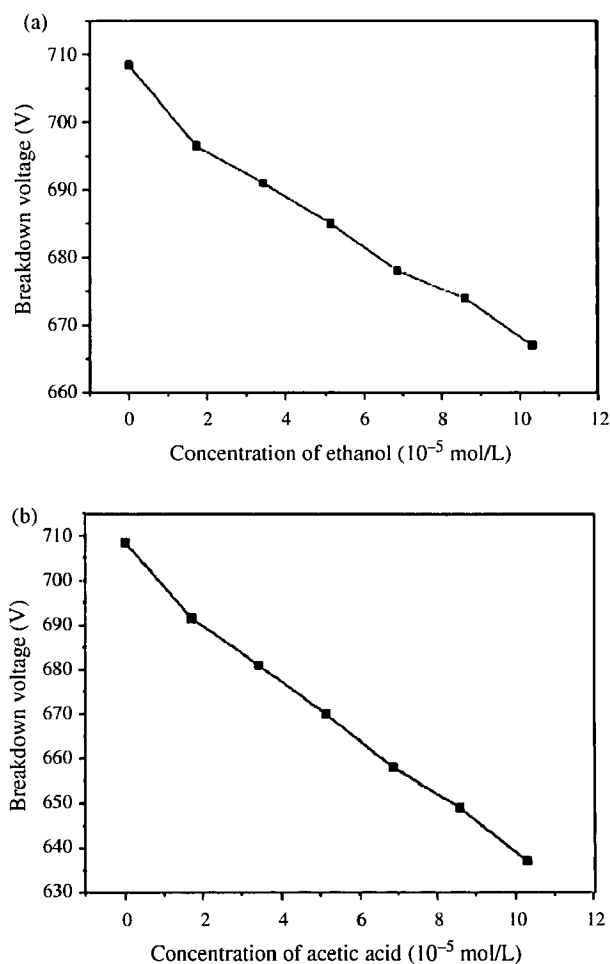


Fig. 8. Breakdown voltage as a linear function of concentration in different gases within a small concentration range: (a) in alcohol; (b) in acetic acid (inter-electrode gap is $50 \mu\text{m}$). Reproduced with permission from [62], X. Chen et al., *Colloids Surf. A* 313–314, 355 (2008). © 2007, Elsevier B. V.

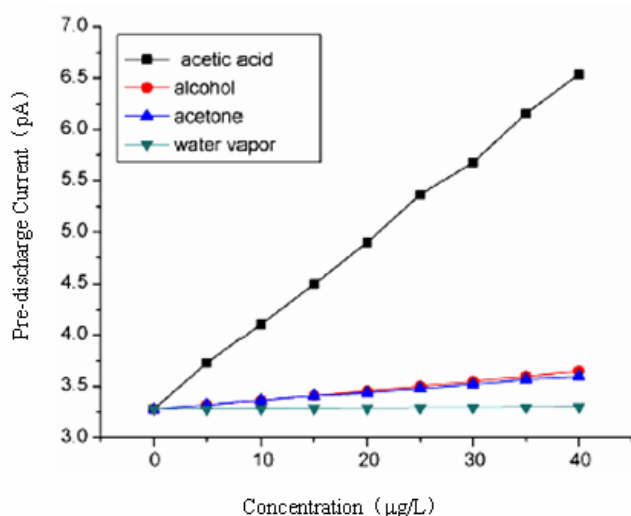


Fig. 9. Pre-discharge current as a linear function of gas concentration within a small concentration range. Reproduced with permission from [62], X. Chen et al., *Colloids Surf. A* 313–314, 355 (2008). © 2007, Elsevier B. V.

shorter life-span of the CNTGIS. Therefore the discharge current density of the CNTGIS is the key factor affecting the life-span of the sensor. It is well established that only non-self-sustaining discharge (viz. pre-discharge) or Townsend self-sustaining discharge produces relatively small discharge current when compared with other discharge states,⁶¹ and this characteristic could potentially lead to longer life-span of the CNTGIS.

9. PERSPECTIVES

The CNTGIS has been considered as a potential micro-nanometre gas sensor due to its high sensitivity to all gases, and especially to trace gas. The studies in the last eight years have indicated that by the use of the CNTGIS, the working voltage is decreased from several thousand volts to several hundreds and even tens volts, and the power consumption of the sensor is also significantly reduced when compared with traditional gas ionization sensors. It has also been found that the CNTGISs with different electrode separations can be used to detect the identity of the component gases directly without the separation of gas mixtures, and consequently offer an advantage over the most precise atmospheric pressure ionization mass spectrometers and chromatographs that separate the mixtures into distinct bands that can then be qualitatively and quantitatively analysed. The other advantage is that the CNTGIS works at room temperature and atmospheric pressure, and thus does not need a vacuum condition for detection. Compared with traditional gas ionization sensors, novel CNTGISs could lower the cost of sensor systems and thereby provide a high performance to cost ratio.

Despite the potential power of CNTGIS as a gas sensing tool, there remain several critical and practical problems that need to be addressed before the novel CNTGIS can be routinely applied in industry. (1) A method should be found to develop a linear relation of discharge voltage and current with gas concentration over a wide concentration range. (2) The repeatability, stability and selectivity of the sensor should be improved.

The coming years are likely to see further advances in CNTGISs in the following directions:

(1) *Quantify gas concentrations over a wide concentration range.* It has been in the last eight years or so that anode-cathode CNTGISs have been studied, but the CNTGIS can not be used on applications because linear discharge characteristics exist only in a small range of gas concentrations. The review in this paper has indicated that it is necessary to probe into the intrinsic characteristics of low power discharges over a wide range of gas concentrations. Moreover, staged-sectional quantitative analyzation methods⁸⁵ for concentrations in different ranges could be used in case low power discharge characteristics are also nonlinear over a wide concentration range.

(2) *Improve the stability of the sensor.* The stability of the sensor is determined by several factors such as the adhesion strength of CNT to substrate material, the stable interelectrode gap, the performance of interelectrode insulation material and the discharge state. Suitable material such as porous silicon can be used as a substrate for CNT to be grown on more tightly and stably than on silicon. Screen printing technology should also be deployed for fabrication of interelectrode glass insulators, leading to a stable electrode separation. Discharge current and discharge voltage should also be decreased to small values, resulting in the generation of a pre-discharge or self-sustaining dark discharge of the CNTGIS, and thereby enabling the decreased thermal consumption of CNT and the longer life-span of CNTGISs.

(3) *Improve the selectivity of the sensor.* CNTGIS array with different electrode separations and data fusion technology are effective methods for eliminating the negative effects of environmental factors and trace impurity gases, and enable better selectivity of CNTGISs, leading to precise detection of gas concentrations.

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