

# Metal–Oxide Nanowire Molecular Sensors

Subjects: [Engineering](#), [Electrical & Electronic](#)

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During the past two decades, one–dimensional (1D) metal–oxide nanowire (NW)-based molecular sensors have been witnessed as promising candidates to electrically detect volatile organic compounds (VOCs) due to their high surface to volume ratio, single crystallinity, and well-defined crystal orientations. Furthermore, these unique physical/chemical features allow the integrated sensor electronics to work with a long-term stability, ultra-low power consumption, and miniature device size, which promote the fast development of “trillion sensor electronics” for Internet of things (IoT) applications.

nanowire

oxide

gas sensor

device

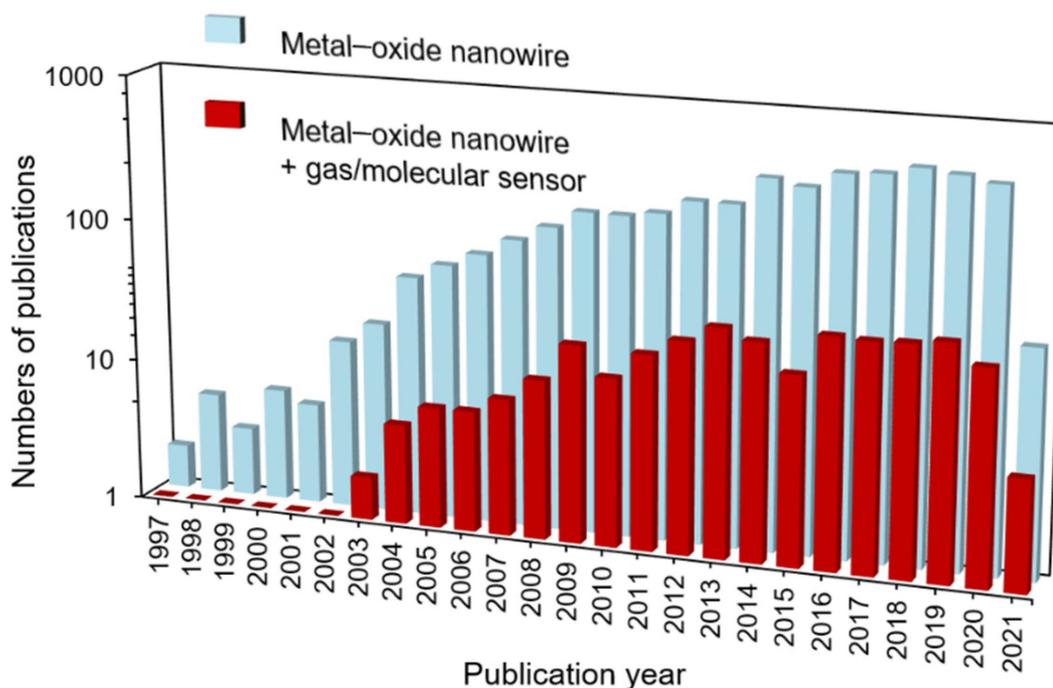
1D nanostructure

sensing mechanism

## 1. Introduction

For the upcoming “trillion sensor electronics” era, molecular sensor and electronic recognition devices, which collect the enormous chemical information as big data from various volatile organic molecules (VOCs), are gaining increasing interests in health care<sup>[1][2][3]</sup>, environment<sup>[1][4][5]</sup>, security<sup>[6][7][8][9]</sup> and agriculture areas<sup>[3][4][10][11]</sup>. Among various molecular sensors, chemiresistive sensors integrated with metal–oxide semiconductor (MOS) nanostructures are of particular interest due to their high sensitivity and fast response<sup>[12][13][14][15][16]</sup>. Especially with the advancement of nanomaterial fabrication technology, a large number of functional MOS nanostructures, such as nanodots<sup>[17][18]</sup>, nanowires<sup>[19][20][21][22]</sup>, nanosheets<sup>[23][24][25]</sup>, and hierarchical nanostructures<sup>[26][27][28]</sup>, are synthesized as building blocks for the fabrication of sensor electronics.

Among these nanostructured forms, 1D MOS nanowires offer an ideal platform for nanoscale sensor integration due to their high surface to volume ratio, comparable sized Debye length, high crystallinity, excellent surface chemical reaction, and low power consumption<sup>[29][30][31]</sup>. Ever since the first report of using the 1D metal–oxide nanostructure as the gas sensor by Comini, great progress has been achieved in the past two decades<sup>[32]</sup>. To date, hundreds of papers have been published on molecular sensors integration based on 1D metal–oxide nanostructures (SnO<sub>2</sub><sup>[29]</sup>, In<sub>2</sub>O<sub>3</sub><sup>[33]</sup>, Fe<sub>2</sub>O<sub>3</sub><sup>[34]</sup>, V<sub>2</sub>O<sub>5</sub><sup>[35]</sup>, CeO<sub>2</sub><sup>[36]</sup>, ZnO<sup>[37]</sup>, WO<sub>3</sub><sup>[38]</sup>, NiO<sup>[39]</sup>, CuO<sup>[40]</sup>, NaNbO<sub>3</sub><sup>[41]</sup>, Zn<sub>2</sub>SnO<sub>4</sub><sup>[42][43]</sup>, CdIn<sub>2</sub>O<sub>4</sub><sup>[44]</sup>, etc.), and the numbers of publications related to nanowires and metal–oxide nanowire sensors can be seen in Figure 1. Meanwhile, to fulfill multiple demands of molecule detection and monitoring, nanowire-based sensor devices with different structures, such as flat<sup>[45]</sup>, suspended<sup>[46]</sup>, bridging<sup>[47]</sup>, and vertical structure<sup>[48]</sup>, have also been addressed. Despite much effort being devoted to advancing the metal–oxide nanowire-based molecular sensor electronics, molecular sensors based on 1D MOS nanowires have not yet been successfully commercialized compared to the MOS film structures.



**Figure 1.** Numbers of publications in the area of nanowires and nanowire sensors from 1997 to 2021 (internet search of the Web of Science on 15 January 2021). Keywords for search: metal-oxide nanowire; metal-oxide nanowire + gas/molecular sensor.

The inherent limitations for the practical applications of the 1D MOS nanowire-based molecular sensors can be summarized as follows:

(1) Lack of effective method for the large-scale synthesis of geometrically uniform single-crystalline nanowires—as is known, the electrical<sup>[49]</sup>, optical<sup>[50]</sup>, thermal<sup>[51]</sup>, and chemical<sup>[52]</sup> properties of nanomaterials are strongly affected by their size and shape due to the quantum confinement effect<sup>[53]</sup>. The electrical conduction becomes more sensitive to the field-effect as the nanowire diameter decreases<sup>[54]</sup>. Until now, although highly crystalline MOS nanowire with various diameters<sup>[55]</sup>, compositions<sup>[28][29][30][31][32][33][34][35][36][37][38][39][40]</sup> and heterostructures<sup>[56][57]</sup> can be grown in vapor phase (physical vapor deposition (PVD)<sup>[58][59]</sup>, pulsed laser ablation deposition (PLD)<sup>[60][61]</sup> and chemical vapor deposition (CVD)<sup>[62][63]</sup>, etc.) and solution phase (hydrothermal<sup>[64][65]</sup> and solvothermal<sup>[66][67]</sup>), the large-scale synthesis of geometry uniform (diameter) nanowires is still a big challenge<sup>[68]</sup>.

(2) Poor reproducibility—it has been demonstrated that the nanowires present a fantastic performance as they are integrated into single nanowire devices<sup>[69]</sup> [69]. For such a kind of device, lithography and sputtering techniques are frequently utilized to design and deposit interdigitated electrodes on randomly distributed nanowires for the device fabrication<sup>[70]</sup>. However, this process is only accessible for fundamental laboratory research, and assembling scalable and controllable nanowires on arbitrary substrates remains a major challenge toward performance reproducible sensor device fabrication<sup>[71]</sup>.

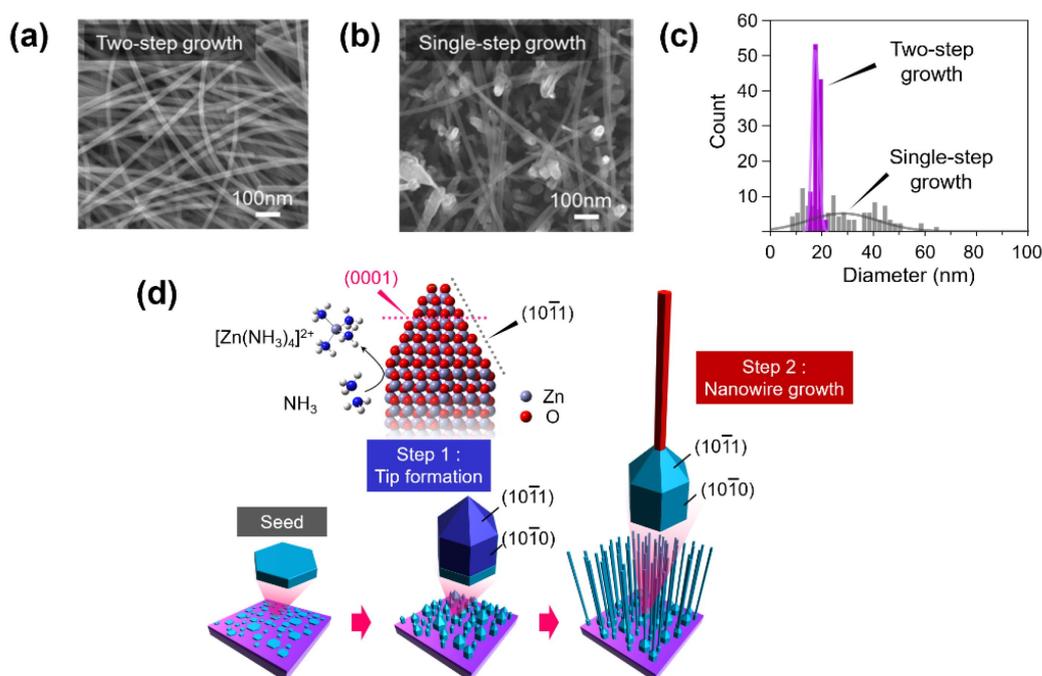
(3) Poor environmental/thermal stability—to deeply exploit the data science from the obtained sensor signal, the long-term stability of the sensor response is highly required for time-series data collection<sup>[72]</sup>. However,

performance degradation usually occurs in the nanowire-based sensor electronics because the surrounding oxygen, water, and contaminants would react with the active nanowire surface as well as the nanowire–electrode contact when the sensors are exposed to ambient air for molecular detection<sup>[47][73][74]</sup>.

(4) Poor sensor selectivity—although the sensitivity of the single nanowire sensor devices has been demonstrated to possess an exponential enhancement as compared with those thin-film devices<sup>[75][76]</sup>, it is still desired to significantly improve the selectivity of nanowire-based sensor electronics. Therefore, further efforts are still encouraged to promote MOS nanowire-based sensor electronics for the IoT applications.

## 2. Prospective towards Metal–Oxide Nanowire Gas Sensor Electronics

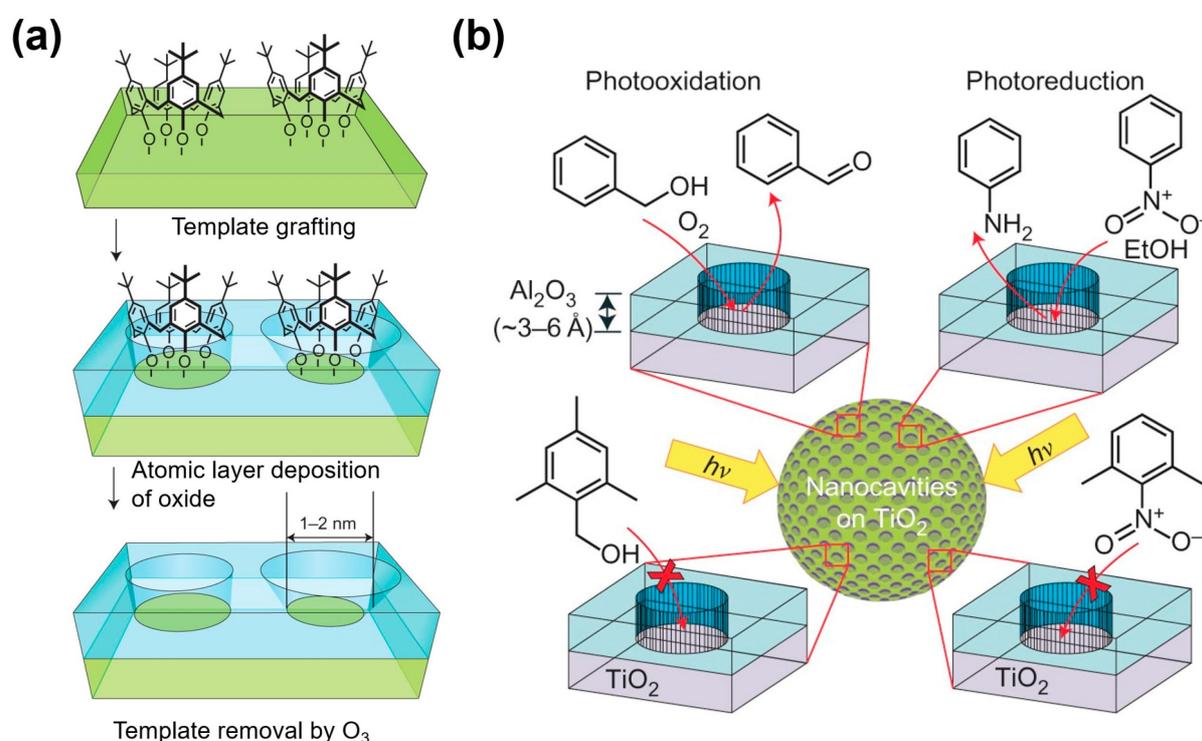
As for the reproducibility of nanowire devices, we should carefully consider how to synthesize nanowires with high uniformity. Template-assisted nanowire growth is considered an alternative approach to achieve a highly ordered nanowire array. However, the complexity of template fabrication, low density of nanowires, and the extensive distribution of nanowire diameter have limited the wide application of such method<sup>[77]</sup>. Recently, Zhao et al. reported a two-step method to fabricate ZnO nanowires with uniformly shaped structures, as shown in Figure 2<sup>[78]</sup>. Firstly, ZnO nanowires of random size are etched by  $\text{NH}_4^+$  as the seed layer, and then, a very similar diameter (average about 17 nm with  $\sigma$  1.3 nm, shown in Figure 2c) of ZnO nanowires are grown in the second step, which significantly increases the reproducibility of metal–oxide nanowire. This unique finding paves the way for the fabrication of nanowires-integrated nanodevices with reliable performance.



**Figure 2.** (a) SEM image of two-step growth ZnO nanowire; (b) SEM image of conventional single-step growth ZnO nanowire; (c) comparison of ZnO nanowire diameter distribution grown by single-step and two-step method;

(d) the mechanism of the synthesis of monodispersed sized ZnO nanowires from randomly sized seeds. Reprinted from reference [78] with permission from the American Chemical Society.

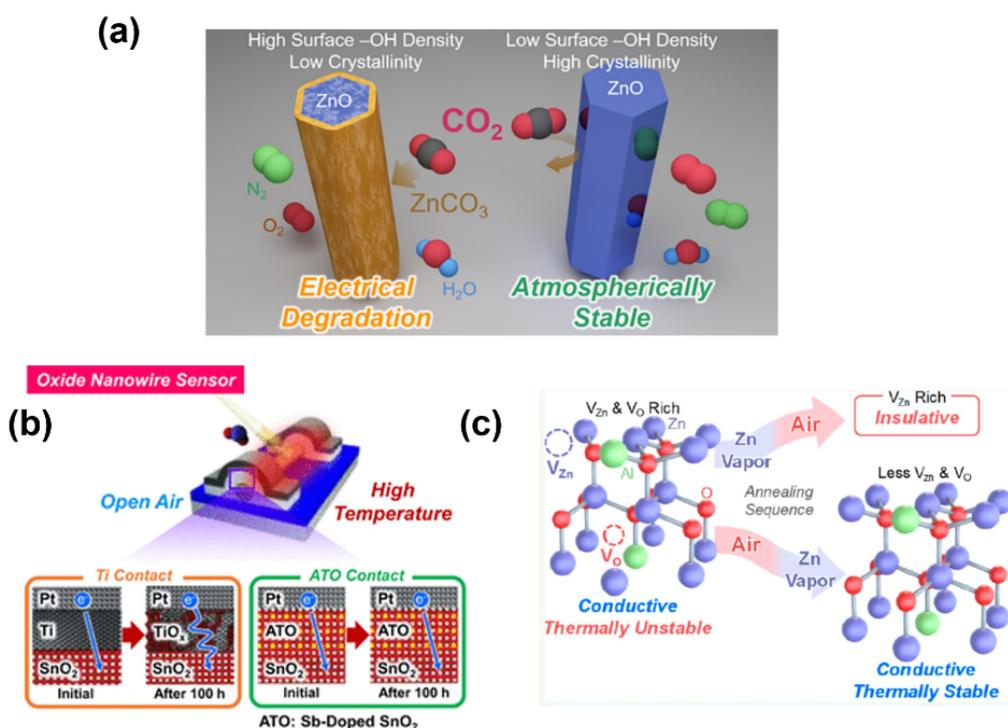
Poor selectivity is a perpetually perplexing problem that limits the wide application of the MOS gas sensor, including the metal–oxide nanowire-based sensors. Inspired by the catalytical chemistry, it is worth considering combining porous materials such as zeolites, metal–organic frameworks, and mesostructured oxides with a metal–oxide nanowire [79][80][81]. Canlas et al. reported a novel method to fabricate molecules imprinted oxide catalyst, and the fabrication process is shown in Figure 3 [82]. Using this structure, the nanocavities can preferentially react with nitrobenzene rather than nitroxybenzene in the photoreduction model and react with benzyl alcohol rather than 2,4,6-trimethylbenzyl alcohol in the photo-oxidation model. This technique can be applied to the metal–oxide nanowire sensor to gigantically improve selectivity due to their preferential interactions with specific VOC molecules, even with a chemically similar structure.



**Figure 3.** (a) The fabrication process of molecules imprinted oxide and (b) the preferential reaction with gas species. Reprinted from reference [82] with permission from the Nature publishing group.

Solving the degradation issues, including nanowire degradation, contact degradation, and electrode degradation, is the only way to achieve long-term stability in nanowire devices. As shown in Figure 4a, Nakamura et al. demonstrated a strategy for achieving the atmospheric electrical stability of ZnO nanowires [74]. Via using a thermal annealing treatment in vacuum/air, the insulating layer induced by the unstable –OH layer on as fabricated ZnO nanowire surface is efficiently eliminated. Such a simple and low-cost method can enhance nanowire atmospheric stability for at least 40 days with stable electrical properties. Meanwhile, Zeng et al. offered a way to overcome the degradation in conventional sensor contact, as shown in Figure 4b [73]. It is found that the nanowire device can obtain good stability for at least over 2000 hours by replacing the easily oxidized contact metal (Ti) with heavily

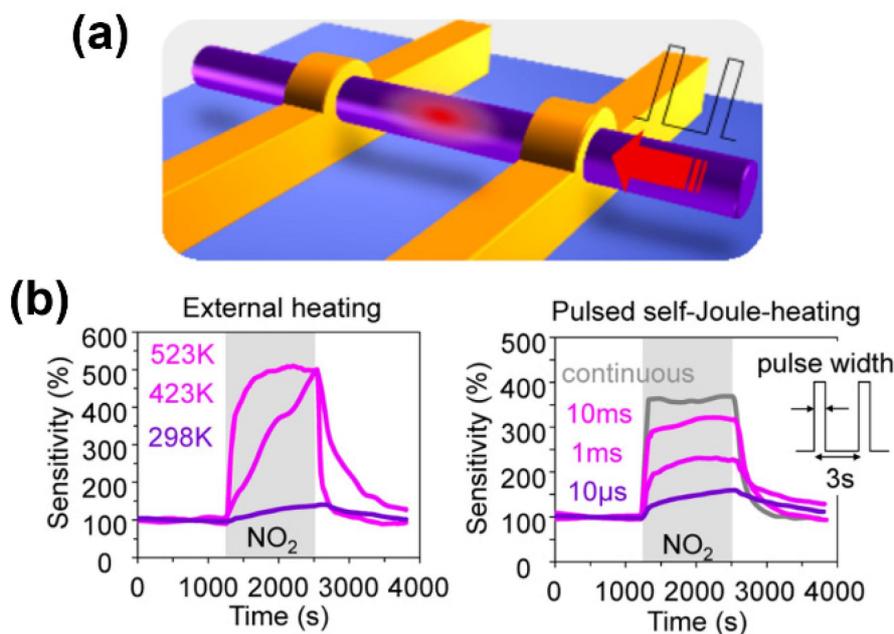
doped metal–oxide (antimony doped tin oxide). Moreover, Yan et al. reported an unusual annealing process on oxide thin films for highly thermal and chemical stability shown in Figure 4c [83][84]. It was indicated that Al-doped ZnO (AZO) nano-thin films could efficiently suppress the inevitable crystal defect formation in the as-fabricated thin film via sequential annealing process under air and Zn vapor atmosphere, resulting in a stable electrical resistivity ( $\sim 10^{-4} \Omega \cdot \text{cm}$ ) in air, even at high temperature (up to 500 °C). This thermally stable thin film can be utilized as electrodes for gas sensors, which obtain stable performance over 250 hours compared to the conventional Ti/Pt contact sensor. Meanwhile, the sequential annealed AZO nano-thin films also show highly chemical stability in buffer solution (pH: 3–11) compared with non-annealed AZO nano-thin films and ZnO thin films. These proposed strategies can successfully suppress the electrical performance degradation of the nanowire devices and have a great potential to be applied to various oxide nanostructures, which would give a foundation for the designing and fabrication of oxide nanomaterial-based IoT sensors with long-term stability.



**Figure 4.** (a) Atmospherically stable ZnO nanowires via using the annealing process. Reprinted from reference [74] with permission from the American Chemical Society; (b) gas sensor with long-term stability via using heavily doped oxide as the contact. ATO, Sb-doped SnO<sub>2</sub>. Reprinted from reference [73] with permission from the American Chemical Society; (c) Al-doped ZnO thin film with thermal stability via using sequential annealing. Reprinted from reference [83] with permission from the American Chemical Society.

With regard to the low power consumption, Meng et al. recently reported an excellent thermal management approach in metal–oxide nanowire sensors via a pulsed self-Joule-heating technique, as shown in Figure 5 [85]. It was found that the thermal conductivity of the device was reduced due to the prohibition of heat dissipation from nanowire to surroundings, and its thermal relaxation times can be decreased down to a microsecond range, while several tens of seconds are needed for conventional MEMS gas sensors. This method enables the reduction in energy consumption down to  $\sim 10^2$  pJ/s and the enhancement of sensitivity for electrical sensing of NO<sub>2</sub> (100 ppb).

This proposed thermal management concept of nanowires in both spatial and time domains offers a strategy for exploring novel functionalities of nanowire-based sensors with high performance.



**Figure 5.** (a) Suspended nanowire device; and (b) comparison between the conventional external heating device and pulsed self-joule-heating device. Reprinted from reference<sup>[85]</sup> with permission from the American Chemical Society.

With the improvement of the metal–oxide nanowire synthesis and device fabrication processes with high uniformity, the integrated nanowire sensor electronics would have a high reproducibility, selectivity, and long-term stability. This would promote the nanowire sensor electronics to be widely used in the IoT applications, such as medicine, food industry, security, and environment protection.

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