

Article

Ultra-Low Loading Single-Atom Pt-Decorated SnO₂ for High-Performance MEMS Hydrogen Sensor

Yuzhou Li ^{1,2}, Xigui Lan ^{1,2}, Yong Yan ¹, Yuanyuan Ge ¹ , Rongrong Jia ³ , Zhili Li ^{1,*}  and Lei Huang ^{2,4,*} 

¹ School of Chemistry and Chemical Engineering, Guangxi University, Nanning 530004, China

² Research Center of Nano Science and Technology, College of Sciences, Shanghai University, Shanghai 200444, China

³ Department of Physics, College of Sciences, Shanghai University, Shanghai 200444, China

⁴ Shanghai Key Laboratory of Chips and Systems for Intelligent Connected Vehicle, Shanghai 200444, China

* Correspondence: lizhili@gxu.edu.cn (Z.L.); leihuang@shu.edu.cn (L.H.); Tel.: +86-21-66137426 (L.H.); Fax: +86-21-66137426 (L.H.)

Abstract

Developing high-response, low-cost H₂ sensors is critical for real-time H₂ monitoring in the new energy era. In this work, ultra-low content (0.07 wt%) single-atom Pt-loaded SnO₂/MEMS H₂ sensors were prepared by an extended two-step annealing method, enabling ppb-level H₂ sensing with low power consumption. At the optimal operating temperature of 201 °C, the sensor showed a response of 55.0 to 100 ppm H₂, which is 9.17 times that of the pure SnO₂ sensor. Compared with SnO₂ sensors loaded with Pt via traditional impregnation, its optimal operating temperature is reduced by nearly 30 °C, and its response value is increased by 45.0. Additionally, the sensor exhibited a fast response time of 2.3 s and a limit of detection as low as 36 ppb. Mechanistic studies reveal that, compared to traditional nanoparticle-modified material, the single-atom Pt-modified material exhibits a higher adsorbed oxygen content and enhanced surface oxidation activity. These results indicate single-atom Pt enhances the active oxygen level, underscoring its critical role in boosting H₂-sensing performance.

Keywords: hydrogen detection; sensor; single atom; Pt; MEMS; ultra-low loading

1. Introduction

With the expanding utilization of hydrogen (H₂) energy, the accurate and reliable detection of hydrogen gas has become increasingly critical for ensuring operational safety and system efficiency. In addition, as the ownership of lithium-ion battery (LIB)-powered electric vehicles continues to rise [1], timely warnings are required when electrolytes leak and thermal runaway (TR) occur in LIBs under extreme conditions (e.g., overcharging, overdischarging, overheating) [2,3]. Given that H₂ is the first gas detected during TR [4], real-time H₂ leakage monitoring is indispensable. Notably, the H₂ concentration in human-exhaled breath is extremely low (1~20 ppm); thus, H₂ sensors need a limit of detection (LOD) at the sub-1 ppm or ppb level [5]. Chemi-resistive gas sensors based on metal oxides (MOS) have emerged as a research focus for H₂ sensing, attributed to their high reliability, low cost, and ease of fabrication [6,7]. Among such sensors, tin dioxide (SnO₂), a typical wide-bandgap n-type semiconductor (bandgap energy E_g = 3.6 eV), stands out as a promising gas-sensing material, featuring high conductivity, chemical stability, and low cost [8,9]. However, pure SnO₂ gas sensors suffer from high operating temperatures and low response. While researchers have improved their gas-sensing performance by loading



Academic Editors: Li Fu and Aiwu Wang

Received: 4 January 2026

Revised: 3 February 2026

Accepted: 4 February 2026

Published: 9 February 2026

Copyright: © 2026 by the authors.

Licensee MDPI, Basel, Switzerland.

This article is an open access article distributed under the terms and conditions of the [Creative Commons Attribution \(CC BY\) license](https://creativecommons.org/licenses/by/4.0/).

noble metals [10–12], such sensors still face challenges including insufficient response, poor integrability, and high cost.

In recent years, single-atom catalysts (SACs) have garnered extensive attention in the field of heterogeneous catalysis owing to their high atomic utilization efficiency and unique physicochemical properties, and their working mechanisms have been successfully extended to the field of gas sensing [13–15]. Compared with nanoparticle- and nanocluster-based catalysts, SACs offer the following advantages: First, SACs can fully activate various active sites to participate in the activation process of oxygen molecules [16,17]. Additionally, SACs can reduce reaction activation energy and enhance the electron transport performance of materials [18]. Owing to the unique coordination environment of SACs, they generally exhibit higher stability and selectivity compared with noble metal nanoparticle catalysts [19,20]. Based on these advantages, SACs have been successfully used to address the shortcomings of MOS-sensing materials. In recent years, noble metal-based single atoms (SAs) (e.g., Pt, Pd, Au) have been widely applied to modify MOS gas-sensing materials such as SnO₂, WO₃, ZnO, and In₂O₃, aiming to enhance response values and selectivity or reduce operating temperatures [21–23]. Koga et al. reported that the incorporation of Pd SAs significantly enhanced the H₂-sensing performance of Co₃O₄ nanoparticle thin films; at a 5% Pd loading, the sensor exhibited high response (~85%) and fast response (~13 s) towards 1000 ppm H₂ [24]. However, most current studies focus on increasing the loading of noble metal single atoms. From a cost perspective, single-atom sensors with low noble metal loading hold greater economic value.

Gas sensors based on micro-electro-mechanical systems (MEMS) represent a cutting-edge direction in current gas sensor research, owing to their numerous advantages such as miniaturized size, low power consumption, easy integrability, and compatibility with intelligent manufacturing [25]. Liang et al. [26] fabricated a ZIF(3)-ZnO/MEMS sensor on MEMS platforms, which was constructed by the in situ growth of ZIF-8 on ZnO. This sensor exhibited a response of 80 towards 25 ppm ethanol at 290 °C. Luo et al. [27] proposed a method to prepare SnO₂ with oxygen vacancy defects via H₂ reduction, and deposited the sensing material onto MEMS chips. The as-prepared MEMS sensor achieved a response of 2.3 towards 6 ppm H₂ at its optimal operating temperature of 250 °C, along with a low LOD of 0.1 ppm. He et al. [28] developed an Nb-doped TiO₂ nanosheet-based MEMS gas sensor for H₂ detection. The sensor showed a response of 2.5 to 1000 ppm H₂ with response/recovery times of 32.5/51 s. However, the development of high-performance H₂ sensors modified with noble-metal single atoms based on MEMS chips is still in its infancy.

Herein, a series of MEMS-based H₂ sensors were fabricated by loading ultra-low content Pt SAs onto SnO₂ nanoparticles via a two-step annealing method. The materials were systematically characterized using transmission electron microscopy (TEM), X-ray diffraction (XRD), ultraviolet–visible spectroscopy (UV–Vis), and X-ray photoelectron spectroscopy (XPS) to analyze their morphology, crystal structure, elemental composition, and valence states. Gas-sensing performance evaluations revealed that the as-prepared sensors exhibited excellent responses to low-concentration H₂. This enhancement was attributed to the improved oxygen activation capability and increased active sites induced by single-atom modification. Compared with the traditional impregnation method, the sensors demonstrated higher response values and lower operating temperatures. Notably, the developed ultra-low content single-atom Pt-loaded SnO₂/MEMS H₂ sensors feature easy integrability and low cost, providing a promising strategy for the fabrication of more economically and high-performance H₂ gas sensors.

2. Results and Discussion

2.1. Structural and Morphological Characterization of Pt/SnO₂

The microscopic morphology of the materials was first analyzed by TEM, with results shown in Figure 1a–d. Figure 1a reveals that SnO₂ consists of nanoparticles with a particle size of ~50–150 nm. After loading with different Pt contents, the morphology and size of SnO₂ show little change, and no obvious Pt particles are observed on the surface (Figure 1b–d). HAADF-STEM images (Figure 1e–g) show small bright spots uniformly distributed on the SnO₂ surface, indicating the formation of isolated Pt SAs [18]. Obvious lattice fringes are visible in Figure 1f, confirming good crystallinity of the material. The interplanar spacing measured from the lattice fringes (inset of Figure 1f) is 0.33 nm, corresponding to the (110) plane of SnO₂ [29]. Notably, the intensity profile of the boxed area in Figure 1g (inset of Figure 1g) exhibits two strong peaks, which correspond to Pt SAs, further confirming that isolated Pt SAs are anchored on the SnO₂ surface [30]. In contrast, for the 0.10 wt%Pt(IM)/SnO₂ sample prepared by simple impregnation, 5 nm Pt nanoparticles can be clearly observed (red circles in Figure S1).

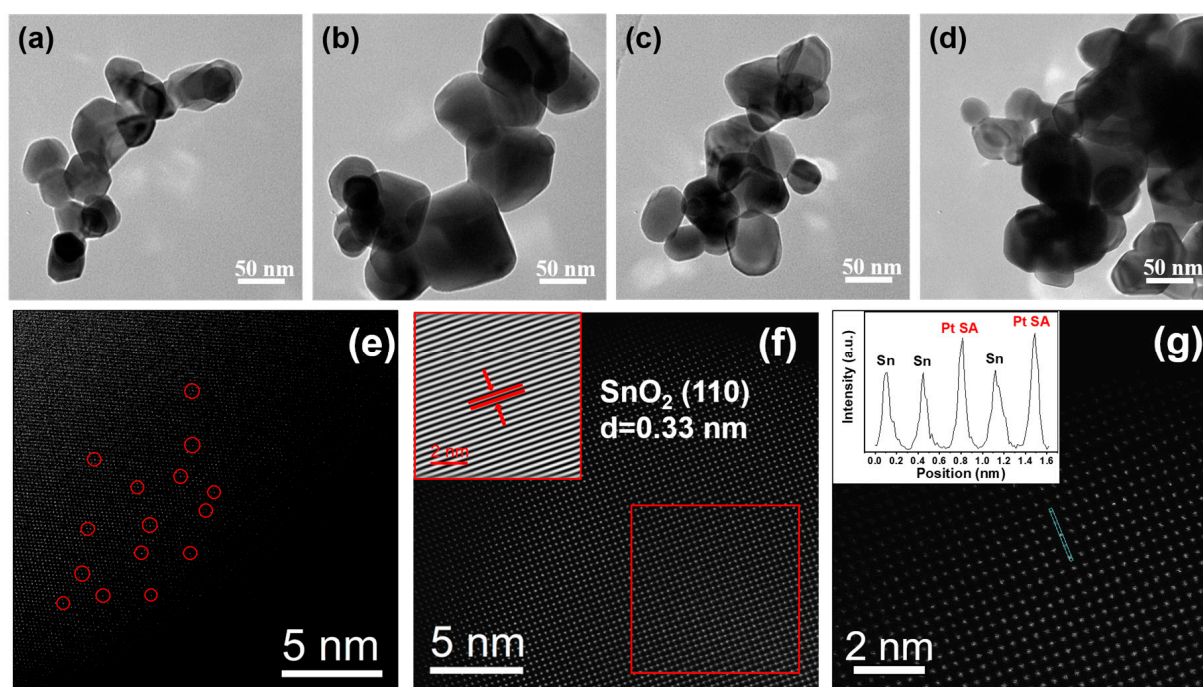


Figure 1. TEM images of different samples: (a) SnO₂, (b) 0.05 wt%Pt/SnO₂, (c) 0.10 wt%Pt/SnO₂, and (d) 0.50 wt%Pt/SnO₂. (e–g) HAADF-STEM images of 0.10 wt%Pt/SnO₂.

XRD was employed to analyze the crystal structure of the synthesized samples, with results presented in Figure 2a. The diffraction peaks of all samples are highly consistent with the standard card of tetragonal rutile phase SnO₂ (PDF#41-1445). Notably, except for the characteristic diffraction peaks of SnO₂, no characteristic diffraction peaks of Pt were detected, which is attributed to the low content and high dispersion of Pt [31]. Regarding the optical absorption properties of the materials, UV-Vis DRS was conducted, as shown in Figure 2b. The loading of Pt not only significantly enhanced the reflectance intensity of SnO₂ but also led to a further increase in the reflectance intensity with the increase in Pt content. This phenomenon further confirms the successful loading of Pt.

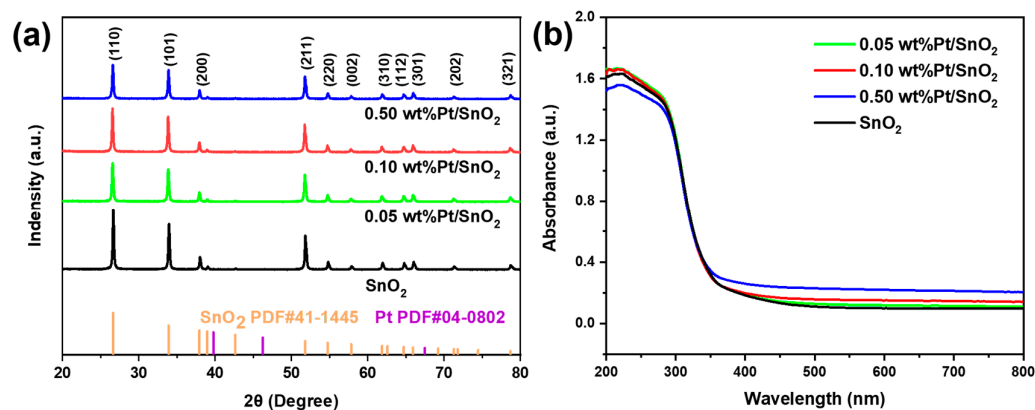


Figure 2. (a) XRD patterns and (b) UV-Vis DRS spectra of Pt/SnO₂ samples with different Pt contents.

To further analyze the surface chemical composition and elemental chemical states of the samples, XPS was employed. Figure 3a presents the full-range high-resolution XPS survey spectrum of 0.10 wt%Pt/SnO₂, in which the characteristic photoelectron peaks of Sn and O elements are clearly identified, confirming the presence of these two elements in the sample. Notably, no obvious Pt 4f characteristic peaks are observed in the full spectral range; this is presumably attributed to the relatively weak signal intensity of the Pt 4f orbital, which is obscured by the strong signals of high-abundance elements [11]. Figure 3b shows the high-resolution Sn 3d XPS spectra. The sharp peaks appearing at 487.10 eV and 495.62 eV are assigned to the Sn 3d_{5/2} and Sn 3d_{3/2} energy levels, respectively. It should be noted that compared with the Sn 3d spectrum of pure SnO₂, the corresponding peak positions of 0.10 wt%Pt/SnO₂ shift toward higher binding energies. This phenomenon indicates that Pt modification facilitates electron transfer from SnO₂ to Pt [32].

As shown in Figure 3c, the O 1s XPS spectrum of 0.10 wt%Pt/SnO₂ can be deconvoluted into three characteristic peaks. The peak at approximately 531.01 eV is assigned to lattice oxygen (O_L) in the SnO₂ lattice; the peak at ~531.69 eV corresponds to defect oxygen (O_V) formed by oxygen vacancy defects on the material surface; and the peak at ~532.88 eV is attributed to chemisorbed oxygen species (O_C) on the material surface [33,34]. Notably, peak area calculations reveal that the O_V content first increases and then decreases with increasing Pt loading. Compared with SnO₂ (19.8%), 0.05 wt%Pt/SnO₂ (15.0%), and 0.50 wt%Pt/SnO₂ (21.6%) (Figure S2a), 0.10 wt%Pt/SnO₂ exhibits the highest O_V content (27.9%), which is consistent with previous literature reports [35]. Meanwhile, the catalytic spillover effect of Pt increases the proportion of O_C to 9.0% [36], which is higher than that of SnO₂ (4.0%), 0.05 wt%Pt/SnO₂ (4.8%), and 0.50 wt%Pt/SnO₂ (7.0%). This indicates that oxygen in 0.10 wt%Pt/SnO₂ exists predominantly in the form of non-lattice oxygen.

Figure 3d presents the high-resolution Pt 4f XPS spectrum of 0.10 wt%Pt/SnO₂ nanoparticles. Notably, as the Pt content increases, the intensity of the Pt 4f peak increases significantly (Figure S2b), confirming the successful loading of Pt onto SnO₂. The high-resolution Pt 4f XPS spectrum can be deconvoluted into two 4f_{7/2} peaks: the peak at 72.80 ± 0.1 eV is attributed to metallic Pt (Pt⁰), while the peaks at 75.47 ± 0.1 eV correspond to Pt²⁺, respectively. The 4f_{5/2} peak at 76.09 ± 0.1 eV is attributed to metallic Pt (Pt⁰), while the peaks at 78.78 ± 0.1 eV correspond to Pt²⁺ [37]. Based on peak area calculations, the percentage contents of Pt⁰ and Pt²⁺ in 0.10 wt%Pt/SnO₂ are 59.0% and 41.0%, respectively. This distribution characteristic of Pt 4f-binding energies indicates that Pt in the sample partly exists in oxidized states (Pt²⁺), which further confirms the atomic-level dispersion of Pt [38].

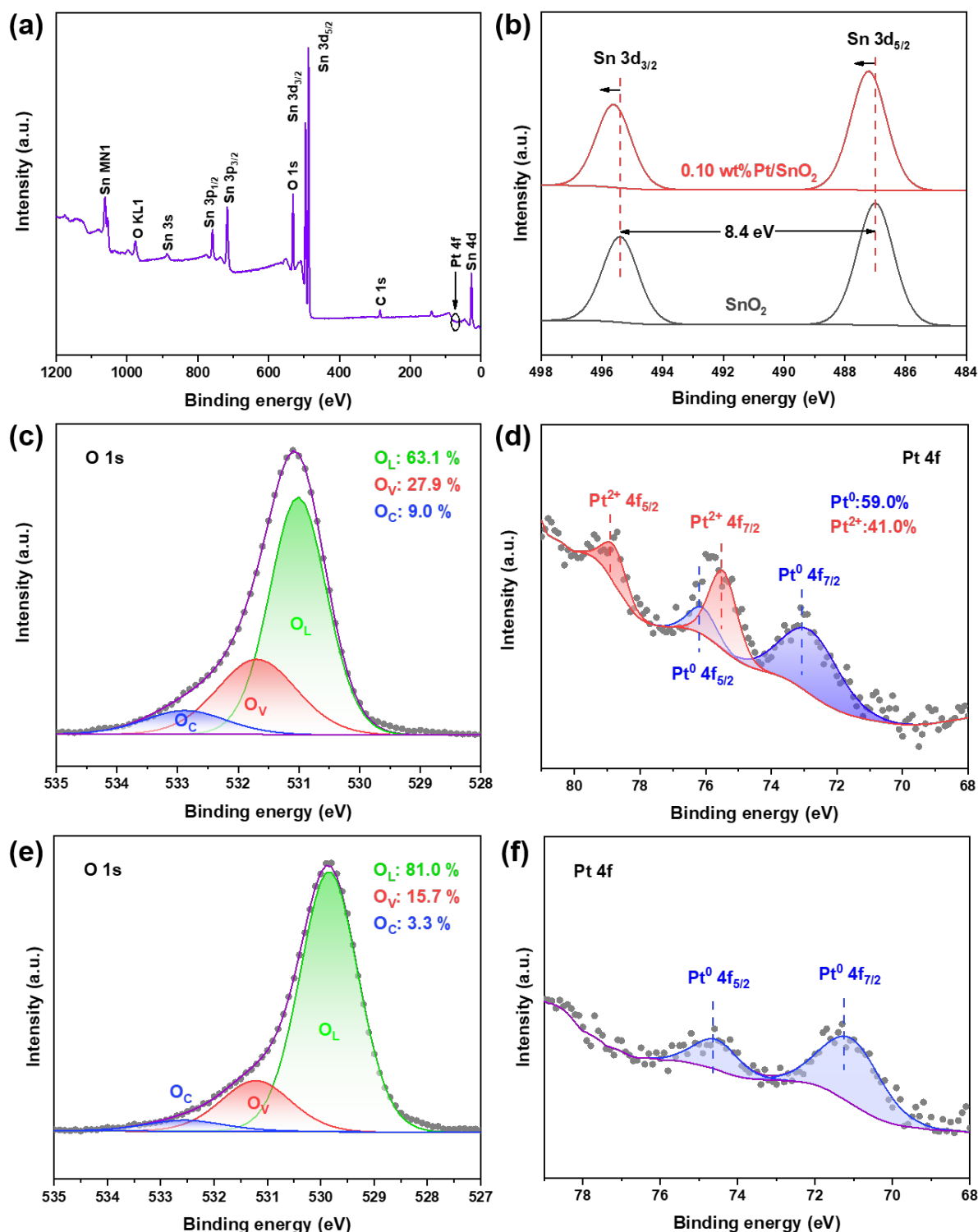


Figure 3. (a) Survey XPS spectrum of 0.10 wt%Pt/SnO₂ nanoparticle. High-resolution XPS spectrum for (b) Sn 3d, (c) O 1s, and (d) Pt 4f of 0.10 wt%Pt/SnO₂ nanoparticles. High-resolution XPS spectrum for (e) O 1s and (f) Pt 4f of 0.10 wt%Pt(IM)/SnO₂ nanoparticles.

Notably, the high-resolution O 1s XPS spectrum of 0.10 wt%Pt(IM)/SnO₂ nanoparticles shows that the relative contents of O_V and O_C are lower than those in 0.10 wt%Pt/SnO₂, accounting for 15.7% and 3.3%, respectively (Figure 3e). Meanwhile, in the high-resolution Pt 4f XPS spectrum of 0.10 wt%Pt(IM)/SnO₂ nanoparticles, no Pt²⁺ peak is observed; instead, Pt exists in the metallic Pt⁰ state (Figure 3f). This result further confirms that Pt in 0.10 wt%Pt(IM)/SnO₂ exists in the form of nanoparticles.

In addition, N₂ adsorption–desorption measurements were conducted to analyze the specific surface area (SSA) of the materials, with results shown in Figure S3. The SSA of 0.10 wt%Pt/SnO₂ is 8.5 m²/g, which is slightly higher than that of pure SnO₂ (8.3 m²/g). The actual Pt loading in different samples was determined via ICP-OES, with results presented in Table S1. The Pt loading increases with the increase in the amount of H₂PtCl₆·6H₂O added. The actual Pt loadings for the 0.05, 0.10, and 0.50 wt% Pt/SnO₂ samples were 0.03%, 0.07%, and 0.19%, respectively. This reduction in actual Pt loading (compared to the designed loading) may be attributed to the following: partial Pt exhibits unstable or ineffective binding with SnO₂ and thus is removed during the water–ethanol washing process.

2.2. Gas-Sensing Performances of Pt-Loaded SnO₂ Sensors

The operating temperature exerts a significant impact on sensor performance [39]. Therefore, the response of sensors toward 100 ppm H₂ under different heating voltages (1.0 V–1.8 V) was first investigated for samples with varying Pt contents (0.05, 0.10, 0.50, and 1.00 wt%), with results presented in Figure 4a. The results indicate that as the heating voltage increases, the sensor response value first increases and then decreases. This trend reflects the typical “volcano-type” relationship between operating temperature and response of metal oxide gas sensors. Meanwhile, with increasing Pt loading, the maximum response value of the sensors also exhibits a “volcano-type” trend: The 0.10 wt%Pt/SnO₂ sensor achieves the optimal gas-sensing performance, showing a response value of around 50 toward 100 ppm H₂ at its optimal operating temperature of 201 °C. In contrast, when the Pt loading increases to 1.00 wt%, the sensor response value drops to about 13. In comparison with the 0.10 wt%Pt(IM)/SnO₂ sensor prepared by the impregnation method, the 0.10 wt%Pt/SnO₂ sensor not only has a lower optimal operating temperature but also exhibits superior gas-sensing performance. Figure S4 shows the response of Pt/SnO₂ with different Pt loading amounts to 100 ppm H₂ at an operating voltage of 1.5 V. With the increase in Pt loading amount, the responses first increase and then decrease.

Figure 4b shows the R_a of each sensor at different operating temperatures. The R_a of all materials decreases as the operating temperature increases. At the same operating temperature, with the increase in Pt content, the R_a of Pt SA-modified sensing materials shows a trend of increasing. Pt has a higher electronegativity than Sn, and Pt SAs capture free electrons from the surface of SnO₂. With the increase in Pt content, it causes a decrease in carriers and an increase in resistance.

Figure 4c further compares the H₂-sensing performance of three sensors: 0.10 wt%Pt/SnO₂, pure SnO₂, and Pt-loaded SnO₂ prepared by simple impregnation (0.10 wt%Pt(IM)/SnO₂). Figure S5 shows that the response value of the 0.10 wt%Pt/SnO₂ sensor to 100 ppm H₂ is 55.0, which is 9.17 times greater than that of pure SnO₂ (response is 6.0). Under the same conditions, the response of 0.10 wt%Pt(IM)/SnO₂ to 100 ppm H₂ is only 6.5, which is far lower than that of 0.10 wt%Pt/SnO₂. This significant performance gap confirms that the loading of Pt in the form of single atoms remarkably enhances the H₂-sensing performance of SnO₂-based sensors.

Further investigation was conducted on the real-time response behavior of the 0.10 wt%Pt/SnO₂ gas sensor toward 10–1000 ppm H₂. Figure 4d shows that the sensor exhibits consistent response and recovery behaviors at various H₂ concentrations, and the response value increases gradually with the increase in H₂ concentration. Figure 4e presents the fitted functional relationship between the response value of the 0.10 wt%Pt/SnO₂ sensor and H₂ concentration. A good linear correlation can be observed between the sensor's response value and H₂ concentration, with a fitting coefficient R² > 99%. The sensor sensitivity (S) is 0.570, and the calculated LOD is 36 ppb. Repeatability and response–

recovery time are also important parameters for evaluating gas sensor performance. For the 0.10 wt%Pt/SnO₂ sensor, the continuous response curves toward 100 ppm H₂ at 201 °C remain essentially consistent over five consecutive tests (Figure 4f), demonstrating excellent reproducibility and accuracy. The dynamic resistance curve of the sensor toward 100 ppm H₂ is shown in Figure 4g. When the adsorption–desorption equilibrium of H₂ gas is reached, the response time and recovery time are 2.3 s and 68.6 s, respectively.

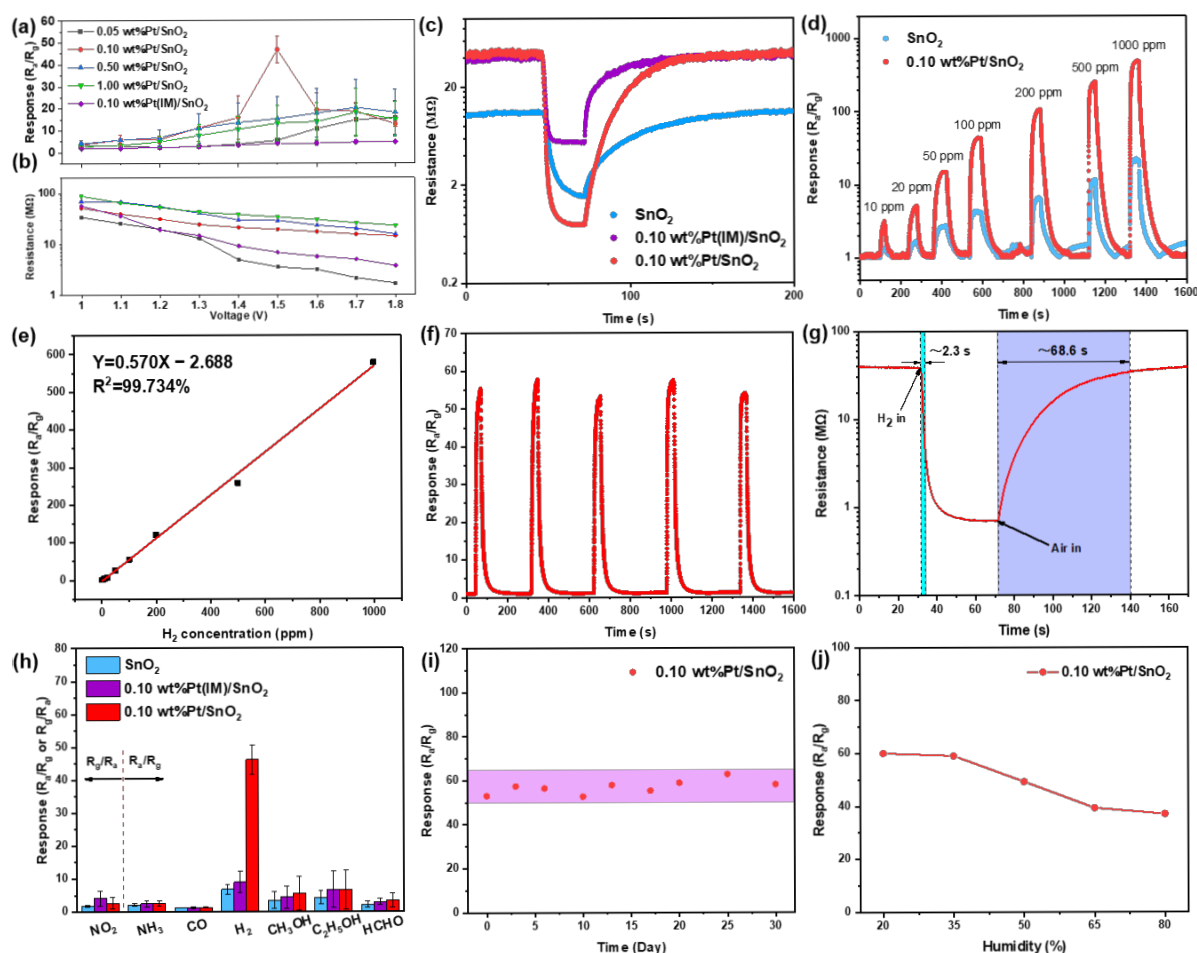


Figure 4. (a) Response to 100 ppm H₂ and (b) R_a of sensors with different Pt contents under different heating voltages. (c) Response transients of sensors based on SnO₂, 0.10 wt%Pt(IM)/SnO₂, and 0.10 wt%Pt/SnO₂ to 100 ppm H₂ at 201 °C. (d) Response of SnO₂ and 0.10 wt%Pt/SnO₂ to 10–1000 ppm H₂ at 201 °C. (e) Fitting function curve for 0.10 wt%Pt/SnO₂ at 201 °C to 10–1000 ppm H₂. (f) Reproducibility of 0.10 wt%Pt/SnO₂ at 201 °C to 100 ppm H₂. (g) Response and recovery times to 100 ppm H₂ for 0.10 wt%Pt/SnO₂ at 201 °C. (h) Selectivity of SnO₂, 0.10 wt%Pt(IM)/SnO₂, and 0.10 wt%Pt/SnO₂ toward different target gases (with NO₂ concentration of 20 ppm and the concentrations of other gases of 100 ppm) at 201 °C. (i) Long-term stability of 0.10 wt%Pt/SnO₂ at 201 °C to 100 ppm H₂. (j) Response of 0.10 wt%Pt/SnO₂ under various RHs to 100 ppm H₂ at 201 °C (the temperature inside the test device is maintained at 20 ± 5 °C).

Multiple gases usually coexist in the environment, so the selectivity of a sensor is also an important indicator for evaluating its performance. Figure 4h shows the sensor's response to several common gases tested at the optimal operating temperature, where the concentration of NO₂ is 20 ppm and the concentration of other gases is maintained at 100 ppm. The responses to methanol and ethanol are 5.57 and 6.62, with selectivity coefficients of 8.26 and 6.95, respectively. Its selectivity coefficients to other gases all exceed 9. Among these, the sensor's response value to CO (a common interfering gas) is only 1.74, and the selectivity coefficient reaches 26.44. It can be seen that the response of the

0.10 wt%Pt/SnO₂ sensor to H₂ is significantly higher than its response to other interfering gases, indicating its excellent selectivity.

To evaluate the long-term stability, the sensor was subjected to a 30-day gas-sensing test with exposure to 100 ppm H₂ (Figure 4i). We conducted 24 h aging to restore the sensor to its original state. The variation range of its response value is approximately $\pm 10\%$, and the response remains relatively consistent, which indicates excellent long-term stability when exposed to H₂. Additionally, the effect of relative humidity on the sensing capability was also studied. As the humidity increases from 20% to 80%, the response of the 0.10 wt%Pt/SnO₂ sensor decreases by 38.4% (Figure 4j), which may be attributed to the competitive adsorption between water molecules and the target gas on the material surface [40]. Thus, humidity compensation is required during its application.

Table 1 summarizes the sensing performance of advanced H₂ sensors modified with Pt. Compared with the reported sensors modified with Pt particles, the SA-based sensor in this study exhibits excellent H₂-sensing characteristics. Notably, compared with the reported Pt-loaded SnO₂ sensor with a Pt loading of 0.1%, the operating temperature of the sensor prepared in this study is reduced by nearly one-third. The low-temperature operation characteristic can reduce the sensor and operating costs. Meanwhile, the LOD is reduced to 36 ppb, whereas that of the reported sensor is only 125 ppb [41]. The high sensitivity and fast response characteristics of the 0.10 wt%Pt/SnO₂ sensor endow it with significant advantages in early-warning scenarios for H₂ leakage.

Table 1. H₂-sensing properties of the different materials in the literature and our present study.

Materials	Metal Oxid Morphology	Noble Metal Morphology	Pt Con. (wt%)	Temp. (°C)	Conc. (ppm)	Resp. (R _a /R _g)	LOD (ppm)	Ref.
Pt/Fe ₂ O ₃ -V _o	Nanosheet	Single-atom	2.59	240	50	~25	0.086	[42]
Pt/ZnO	Pencil-like	Nanoparticle	~2.4	150	100	~2.8	10	[43]
Pt/SnO ₂	Nanofiber	Nanoparticle	0.1	300	2.5	16.6	0.125	[41]
Pt/Sn/5%Co	Nanoparticle	Nanoparticle	1	300	100	57.9	10	[44]
Pt/SnO ₂	Nanoparticle	Nanoparticle	~1.3	400	5000	50	1	[45]
PtSn ₂ -rGO-SnO ₂	Nanoparticle	Nanoparticle	~0.82	175	500	20.25	1	[46]
0.10 wt%Pt/SnO ₂	Nanoparticle	Single-atom	0.1	201	100	55.0	0.036	This work

2.3. Sensing Mechanism

For n-type MOS semiconductors such as SnO₂, a widely accepted gas-sensing mechanism lies in the resistance change of the material caused by gas adsorption and reaction. When the sensing material is exposed to air, O₂, due to its high electron affinity, captures electrons from the conduction band of the material to form surface-adsorbed oxygen (Figure 5, Equations (1)–(3)), where “gas” and “ads” represent gaseous oxygen and adsorbed oxygen, respectively. This process reduces the electron concentration in the material, forms an electron depletion layer (EDL), and thus significantly increases the initial resistance of the material [47,48]. When H₂ is introduced, H₂ may be adsorbed on the SnO₂ surface and dissociated into hydrogen atoms. The adsorbed hydrogen atoms react with O[−] ions, returning the electrons captured by O[−] to the SnO₂ (Equations (4) and (5)). As a result, the depletion layer shrinks and the resistance decreases accordingly (Figure 5a).



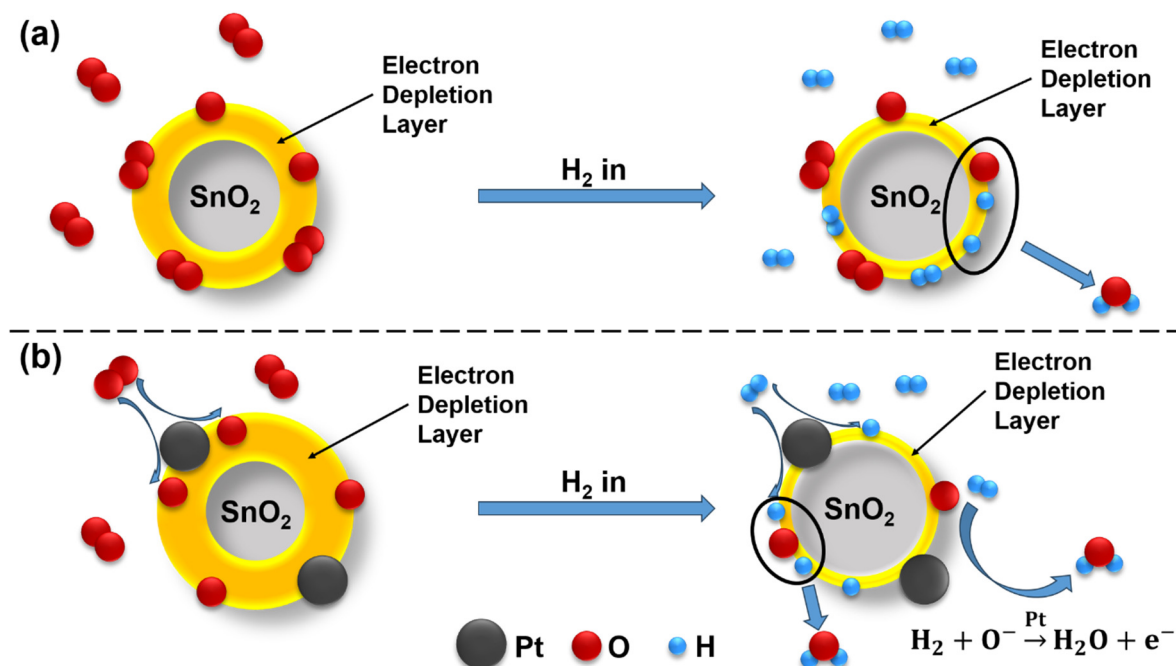
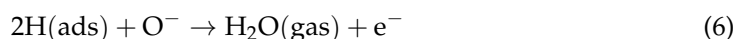


Figure 5. Schematic diagrams for the H_2 -sensing mechanism of (a) SnO_2 and (b) 0.10 wt%Pt/ SnO_2 .

In the above process, the activation and reaction of H_2 and O_2 facilitate the change in the depletion layer, which is also a key process for strong chemical sensitization. Previous XPS results show that Pt SAs in 0.10 wt%Pt/ SnO_2 coordinate with O to form Pt–O bonds, with content of 41.0%. Pt atoms are dispersed in the SnO_2 lattice by substituting Sn atoms, and the Pt–O bonds around them are weaker than Sn–O bonds [49]. When H_2 is present, H_2 preferentially reacts with lattice oxygen near Pt, leading to the breakage of Pt–O bonds and the generation of oxygen defects. These oxygen defects diffuse to the sensing layer through the interface between the Pt– SnO_2 layer and the SnO_2 -sensing layer, altering the electronic state of SnO_2 and increasing the carrier density, thereby significantly improving the H_2 sensitivity of the sensor modified with Pt SAs (Figure 5b). In addition, this process is reversible: when H_2 disappears, Pt can be re-oxidized by air to recombine with oxygen, and the supplemented oxygen atoms diffuse back to the sensing layer, reducing the number of carriers and restoring the sensor to its initial state. This ensures the stability and repeatability of sensing [50].

The oxidation activity of surface species was further characterized by H_2 -TPR. Figure 6a shows the reduction curves of 0.10 wt%Pt/ SnO_2 , 0.10 wt%Pt(IM)/ SnO_2 , and SnO_2 in the temperature range of 100–800 °C. For all samples, the strong peaks in the range of 500–800 °C are attributed to the reduction in SnO_2 lattice oxygen [51], while the peaks below 350 °C may originate from the reduction in chemisorbed oxygen ions and hydroxyl groups on the sample surface. Notably, as shown in the enlarged curve in Figure 6b, the Pt-loaded samples exhibit weak peaks in the low-temperature region. Normally, these weak peaks reflect the surface oxidation activity of the materials: the stronger the reduction peak, the higher the surface oxidation activity. This confirms that Pt introduces new low-temperature active sites and reduces the dissociation activation energy of H_2 [52]. Here, the low-temperature peak positions of 0.10 wt%Pt/ SnO_2 and 0.10 wt%Pt(IM)/ SnO_2 are close, indicating that there is little difference in H_2 dissociation ability between the two samples.

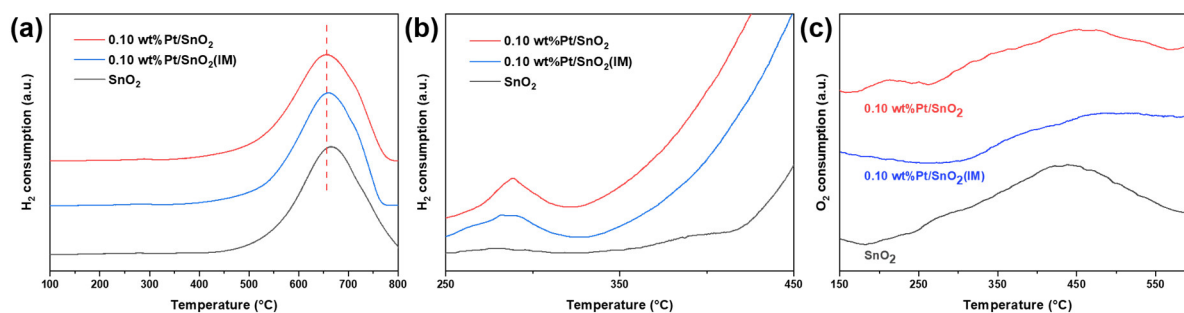


Figure 6. (a) H₂-TPR spectra of 0.10 wt%Pt/SnO₂, 0.10 wt%Pt(IM)/SnO₂, and SnO₂. (b) Enlarged H₂-TPR spectra in the temperature range of 250–450 °C. (c) O₂-TPD spectra of 0.10 wt%Pt/SnO₂, 0.10 wt%Pt(IM)/SnO₂, and SnO₂.

In the O₂-TPD results (Figure 6c), peaks below 300 °C are usually attributed to the desorption of surface-adsorbed oxygen, peaks between 300 °C and 500 °C correspond to the desorption of oxygen at oxygen vacancies, and peaks around 500 °C are desorption peaks associated with lattice oxygen desorption [53]. All three materials exhibit desorption peaks in the range of 450 °C to 500 °C (corresponding to lattice oxygen desorption). However, 0.10 wt%Pt/SnO₂ shows an obvious desorption peak at approximately 200 °C, indicating that compared with 0.10 wt%Pt(IM)/SnO₂ and SnO₂, 0.10 wt%Pt/SnO₂ has a stronger oxygen release performance. This result demonstrates that compared with the traditional nanoparticle-based modification method, Pt SA modification effectively enhances the oxygen dissociation capacity of the material, thereby improving its overall gas-sensing performance.

The loading of Pt SAs leads to an increase in adsorbed oxygen, significantly enhancing the material's oxygen release capacity. The surface of 0.10 wt%Pt/SnO₂ adsorbs more active oxygen species, demonstrating that Pt SAs are more favorable for oxygen adsorption and activation than the conventional impregnation method. XPS results further confirm that the high catalytic activity of Pt promotes the dissociation of O₂ molecules in air into adsorbed oxygen (O[−](ads)) [37]. XPS data show that the adsorbed oxygen content of 0.10 wt%Pt/SnO₂ (9.0%) is 2.25 times that of SnO₂ (4.0%) and significantly higher than that of 0.10 wt%Pt(IM)/SnO₂ (3.3%). These results indicate that compared with conventional noble metal loading via impregnation, single-atom loading significantly increases the adsorbed oxygen content of the material and enhances its oxygen activation capacity. This explains why the optimal operating temperature of the single-atom-modified material is significantly lower than that of the unloaded and impregnated samples.

It is worth noting that in this study, Pt is dispersed on the SnO₂ surface in the form of single atoms, and its role is primarily manifested as surface catalytic enhancement, rather than significantly altering the bulk electronic structure of SnO₂. This observation is consistent with recent studies on the interfacial electronic behavior of SnO₂ [54]. This result suggests that in SnO₂-based functional materials, interface engineering often has a far greater impact on surface reactivity than on modulating the bulk electronic structure. In our work, Pt SA increases the content of O_C and O_V. UV–Vis spectra show minimal shift in SnO₂ absorption edges, confirming that bulk electronic properties remain stable. Meanwhile, the R_a of the sensing material and its sensing performance exhibit distinct variation trends: R_a decreases only with the increase in heating voltage, whereas the response value increases first and then decreases. Thus, the enhanced sensing response originates predominantly from surface catalytic processes rather than bulk conductivity changes, aligning with the interfacial-dominant behavior reported in the aforementioned reference [54].

In summary, via substitutional doping Pt SAs, the Pt-O bonds create conditions for H_2 to preferentially react with adjacent lattice oxygen: this reaction causes the breakage of Pt-O bonds and the generation of oxygen defects. These defects not only diffuse to the sensing layer to increase carrier density but also provide channels for the transport of subsequent reactive species. Meanwhile, due to the single-atom dispersion characteristic of Pt SAs, they not only introduce new low-temperature active sites but also improve the material's oxygen activation efficiency and active oxygen level. The dissociated H_2 species can quickly contact active oxygen through oxygen defect channels, thereby promoting the oxidation reaction of adsorbed H_2 and increasing H_2 conversion efficiency. In conclusion, Pt SAs enhance the material's oxygen activation ability and active oxygen amount, promote the activation of H_2 and its reaction with active oxygen, and increase the number of active sites of the material. Ultimately, 0.10 wt%Pt/SnO₂ exhibits higher H_2 response, lower LOD, and shorter response time.

3. Materials and Methods

3.1. Chemicals

Tin dioxide (SnO₂, 99.9%) was purchased from Shanghai McLean Biochemical Technology Co., Ltd. (Shanghai, China), Chloroplatinic acid ($H_2PtCl_6 \cdot 6H_2O$, AR). Terpeneol mixture of isomers (C₁₀H₁₈O, CP), ethanol (C₂H₅OH, $\geq 99.7\%$), methanol (CH₃OH, $\geq 99.7\%$) and Formaldehyde (HCHO, 40%) were sourced from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). No further purification was carried out on the reagents, and the water used was deionized.

3.2. Synthesis of Pt-Loaded SnO₂

The typical synthesis process for Pt-loaded SnO₂ with varying Pt contents is as follows (Figure S6): 1.0 g of SnO₂ was weighed into a beaker, and different volumes of chloroplatinic acid solution were added according to the designed Pt loading. A volume of 50 mL absolute ethanol was added as the solvent, and the mixture was sonicated in an ultrasonic cleaner for 10 min to ensure uniform dispersion of the chloroplatinic acid impregnation solution in SnO₂. The dispersed sample was then stirred in a constant-temperature water bath at 70 °C until complete evaporation of the solvent, followed by drying overnight in an oven at 70 °C. The dried sample was transferred to a crucible and calcined in a muffle furnace at 300 °C for 5 h with a heating rate of 5 °C/min, followed by natural cooling. This heat treatment ensured firm anchoring of the Pt precursor on the SnO₂ surface [55]. Subsequently, the powder was repeatedly washed with water-ethanol mixture three times, filtered, and dried overnight at 70 °C. After drying, the powder was placed in a crucible and calcined in a muffle furnace at 500 °C for 5 h with a heating rate of 5 °C/min, then collected after natural cooling. This heat treatment decomposed the Pt precursor, removed Pt ligands, and finally formed single atoms [55]. The resulting Pt-loaded SnO₂ with Pt mass percentages of 0.05, 0.10, 0.50, 1.00, and 1.50 wt% were denoted as X wt%Pt/SnO₂ (where X represents the mass fraction of Pt). For comparison, the 0.10 wt%Pt-loaded SnO₂ sample obtained after simple impregnation was calcined in a muffle furnace under a 4% H_2 atmosphere at 300 °C for 2 h with a heating rate of 10 °C/min; the resulting sample was denoted as 0.10 wt%Pt(IM)/SnO₂ (IM stands for impregnation method).

3.3. Characterization of Sensing Materials

The crystal structure and phase composition of the materials were analyzed by X-ray diffraction (XRD, Bruker D2 PHASER, Mannheim, Germany) with a Cu K α target ($\lambda = 0.154184$ nm) operated at 30 kV and 10 mA. The scanning range was 20° to 80° with a scanning rate of 10° min⁻¹. The morphology and microstructure of the mate-

rials were observed using transmission electron microscopy (TEM, JEOL JEM-2100Plus, Akishima, Japan) and aberration-corrected transmission electron microscopy (AC-TEM, JEM-ARM300F, JEOL, Akishima, Japan). High-angle annular dark-field (HAADF) images from AC-TEM enabled direct visualization of single atoms [16]. Optical absorption was measured using a UV–visible diffuse reflectance spectrometer (UV–VIS DRS, Shimadzu UV-3600i Plus, Kyoto, Japan) with BaSO₄ as the reflectance standard. X-ray photoelectron spectroscopy (XPS, Thermo Scientific K-Alpha, Waltham, MA, USA) was employed to analyze the elemental composition and valence state distribution, with calibration against the C 1s standard peak (284.8 eV). The actual Pt loading in different samples was determined by inductively coupled plasma optical emission spectrometry (ICP-OES, Agilent 5110, Santa Clara, CA, USA). A fully automatic specific surface area and porosity analyzer (Micromeritics ASAP 2460, Norcross, GA, USA) was used to characterize the specific surface area and pore structure. The specific surface area was calculated using the Brunauer–Emmet–Teller (BET) model, and the pore size distribution was derived via the Barrett–Joyner–Halenda (BJH) algorithm. Temperature-programmed reduction (TPR, VDSorb-92i, Quzhou, China) and temperature-programmed desorption (TPD, WFS-3015, Tianjin, China) were performed to analyze the surface active oxygen species and oxidation activity of the materials.

3.4. Preparation and Performance Testing of MEMS Sensors

The MEMS chip consists of interdigital electrodes, an isolation layer, a heating electrode, and a support layer (Figure S7). Both the length and width of the central active area are 150 μm . The heating electrode provides the required operating temperature for the gas sensor, while the interdigital electrodes detect resistance changes in the gas-sensing material. The relationship between heating voltage and temperature is shown in Figure S8, enabling control of the sensor's operating temperature by adjusting the heating voltage (V). The relationship between power consumption and heating voltage is depicted in Figure S9. Notably, at a heating voltage of 1.5 V, the sensor exhibits a power consumption of only 22.0 mW.

The fabrication process of the MEMS gas sensor is as follows: First, 15 mg of the sample was weighed and placed into an agate mortar, followed by the addition of 3 drops of turpentine. The mixture was then ground until the powder was uniformly dispersed in the liquid. A drawing pen was used to dip an appropriate amount of the mixture, which was gently coated onto the MEMS chip. After coating, the chip was placed into a square crucible and transferred to a muffle furnace, where it was heat-treated at 250 $^{\circ}\text{C}$ for 2 h with a heating rate of 2 $^{\circ}\text{C min}^{-1}$. Subsequently, the MEMS chip with the sample was placed on the testing equipment and aged at a heating voltage of 1.8 V for 24 h to enhance its stability.

The gas-sensing performance tests were performed with reference to previous work [56]. A Lingpan Electronic Gas-Sensing Tester (Shanghai Lingpan Electronic Technology Co., Ltd., Shanghai, China, LabVIEW version number 17.0.1f1) was used, adopting the static distribution method. The ambient temperature was maintained at 20 ± 5 $^{\circ}\text{C}$, and the relative humidity (RH) was $35 \pm 5\%$ RH. The volume of the experimental apparatus was 195 mL, and different gas concentrations were obtained via the standard gas dilution method. Standard gases (NO₂, H₂, CO, NH₃, NO) were purchased from Shanghai Wetry Standard Gas Analysis Technology Co., Ltd. (Shanghai, China). Gases including CH₃OH, HCHO, and C₂H₅OH were prepared using a self-made gas generation device (Figure S10). Specifically, a micropipette was used to draw the corresponding liquid, which was then dropped onto a heating plate; the liquid was evaporated by heating to achieve the desired concentration. The volume of the injected liquid, denoted as V_x (mL), could be calculated using Equation (7), where V is the volume of the test chamber (mL), C is the vapor concentration of the liquid (ppm), M is the molar mass of the liquid (g/mol), d is the

density of the liquid (g/cm^3), p is the purity of the liquid, T_r is the room temperature ($^{\circ}\text{C}$), and T_b is the temperature inside the test chamber ($^{\circ}\text{C}$).

$$V_x = \frac{V \times C \times M}{22.4 \times d \times p} \times 10^{-9} \times \frac{273+T_r}{273+T_b} \quad (7)$$

To create environments with different humidity levels (20% RH to 80% RH), low humidity was controlled by adjusting the amount of silica gel in the gas-sensing test chamber, while high humidity was achieved using a humidifier. The humidity level was calibrated with a LE502-WH hygrometer (DELI, Ningbo, China). The temperature inside the test device is maintained at 20 ± 5 $^{\circ}\text{C}$.

The limit of detection (LOD) can be calculated using Equations (8)–(10):

$$\text{LOD} = 3\text{RMS}/\text{Slope} \quad (8)$$

$$\text{RMS} = \sqrt{\frac{V_x^2}{N}} \quad (9)$$

$$V_x^2 = \sum (y_i - y)^2 \quad (10)$$

Herein, RMS refers to the root mean square of the response signal. The Slope denotes the slope of the straight line obtained by linear fitting of the sensor's response data to 10–1000 ppm H_2 . y_i represents the measured response data at the baseline in the absence of the target gas, while y is the optimal response value without the target gas ($y = 1$). N is the number of data points, which is 50, and the baseline duration is 5 s. The data of the 10–1000 ppm H_2 concentration gradient used for LOD calculation were obtained from the calibrated data of multiple sensors. The response of the gas sensor is defined differently for oxidizing and reducing gases: $R = R_g/R_a$ for oxidizing gases and $R = R_a/R_g$ for reducing gases, where R_a is the sensor resistance in air, and R_g is the sensor resistance in the target gas atmosphere. The response time and recovery time are defined as the time required for the resistance change to reach 90% of the total resistance change [57].

4. Conclusions

In this study, atomically dispersed Pt-loaded SnO_2 with ultra-low loading was successfully prepared, and MEMS H_2 gas sensors were also fabricated. Among the samples, the 0.10 wt%Pt/ SnO_2 nanoparticles exhibit the optimal comprehensive gas-sensing performance, with a response of 55.0 toward 100 ppm H_2 at 201 $^{\circ}\text{C}$. Compared with pure SnO_2 and 0.10 wt%Pt(IM)/ SnO_2 , the sensor modified with Pt SAs has an optimal operating temperature reduced by nearly 30 $^{\circ}\text{C}$, and its response value is significantly higher than that of 0.10 wt%Pt(IM)/ SnO_2 (6.5). In addition, the sensor also demonstrates excellent H_2 selectivity, a LOD as low as 36 ppb, and good long-term stability. The enhanced sensing performance of the single-atom-modified material can be attributed to the role of Pt SAs in promoting O_2 dissociation, improving the oxygen activation capacity of the material. This work provides a potential approach for the development of low-cost and low-power H_2 sensors, and holds broad application prospects in the new energy era where the demand for H_2 detection is increasingly growing.

Supplementary Materials: The following supporting information can be downloaded at <https://www.mdpi.com/article/10.3390/molecules31040607/s1>. Figure S1. TEM image of 0.10 wt%Pt(IM)/ SnO_2 . Figure S2. (a) High resolution XPS spectra for O 1s of SnO_2 , 0.05 wt%Pt/ SnO_2 and 0.50 wt%Pt/ SnO_2 , (b) High resolution XPS spectra for Pt 4f of 0.05 wt%Pt/ SnO_2 and 0.50 wt%Pt/ SnO_2 . Figure S3. N_2 adsorption isotherms and specific surface areas of SnO_2 and 0.10 wt%Pt/ SnO_2 . Figure S4. Response of Pt/ SnO_2 with different Pt loading amounts to 100 ppm H_2 at an operating voltage of 1.5 V.

Figure S5. The response value of sensors to 100 ppm H₂. Figure S6. Preparation flowchart of X wt%Pt/SnO₂ material. Figure S7. Component diagram of MEMS chip. Figure S8. Plot of heating voltage versus working temperature of the MEMS chips. Figure S9. Plot of heating voltage versus power consumption of the MEMS chips. Figure S10. Gas generation device. Table S1. ICP-OES test results of Pt/SnO₂ samples with different Pt loadings.

Author Contributions: Y.L.: Investigation, Data curation, Validation, Writing—original draft, Writing—review and editing. X.L.: Investigation. Y.Y.: Investigation. Y.G.: Resources, Funding acquisition. R.J.: Data curation, Project administration. Z.L.: Methodology, Supervision, Writing—review & editing, Resources. L.H.: Conceptualization, Supervision, Methodology, Writing—review and editing, Funding acquisition. All authors have read and agreed to the published version of the manuscript.

Funding: This work was financially supported by Shanghai Key Laboratory of Chips and Systems for Intelligent Connected Vehicle (N.99-0144-25-314) and Jiangsu Shuangyi Intelligent Technology Co., Ltd. (2022450001000960).

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: The original contributions presented in this study are included in the article/Supplementary Material. Further inquiries can be directed to the corresponding authors.

Conflicts of Interest: The manuscript received research grants from Jiangsu Shuangyi Intelligent Technology Co., Ltd. The sponsors had no role in the design, execution, interpretation, or writing of the study.

References

- Bastida-Molina, P.; Hurtado-Pérez, E.; Peñalvo-López, E.; Moros-Gómez, M.C. Assessing transport emissions reduction while increasing electric vehicles and renewable generation levels. *Transp. Res. Part D Transp. Environ.* **2020**, *88*, 102560. [\[CrossRef\]](#)
- Huang, P.; Wang, Q.; Li, K.; Ping, P.; Sun, J. The combustion behavior of large scale lithium titanate battery. *Sci. Rep.* **2015**, *5*, 7788. [\[CrossRef\]](#) [\[PubMed\]](#)
- Ren, D.; Feng, X.; Lu, L.; He, X.; Ouyang, M. Overcharge behaviors and failure mechanism of lithium-ion batteries under different test conditions. *Appl. Energy* **2019**, *250*, 323–332. [\[CrossRef\]](#)
- Jin, Y.; Zheng, Z.; Wei, D.; Jiang, X.; Lu, H.; Sun, L.; Tao, F.; Guo, D.; Liu, Y.; Gao, J.; et al. Detection of Micro-Scale Li Dendrite via H₂ Gas Capture for Early Safety Warning. *Joule* **2020**, *4*, 1714–1729. [\[CrossRef\]](#)
- Zhong, A.; Shen, B.; Wang, T.; Jin, H.; Xie, Y.; Zhang, D.; Li, H.; Liu, H.; Luo, J.; Fan, P. Ultrafast H₂ gas nanosensor for ppb-level H₂ gas detection based on GaN honeycomb nanonetwork. *Sens. Actuators B Chem.* **2021**, *329*, 129079. [\[CrossRef\]](#)
- Shaik, R.; Kampara, R.K.; Kumar, A.; Sharma, C.S.; Kumar, M. Metal oxide nanofibers based chemiresistive H₂S gas sensors. *Coord. Chem. Rev.* **2022**, *471*, 214752. [\[CrossRef\]](#)
- Xi, J.; Jung, H.S.; Xu, Y.; Xiao, F.; Bae, J.W.; Wang, S. Synthesis Strategies, Catalytic Applications, and Performance Regulation of Single-Atom Catalysts. *Adv. Funct. Mater.* **2021**, *31*, 2008318. [\[CrossRef\]](#)
- Sukunta, J.; Wisitsoraat, A.; Tuantranont, A.; Phanichphant, S.; Liewhiran, C. Highly-sensitive H₂S sensors based on flame-made V-substituted SnO₂ sensing films. *Sens. Actuators B Chem.* **2017**, *242*, 1095–1107. [\[CrossRef\]](#)
- Barsan, N.; Simion, C.; Heine, T.; Pokhrel, S.; Weimar, U. Modeling of sensing and transduction for p-type semiconducting metal oxide based gas sensors. *J. Electroceramics* **2010**, *25*, 11–19. [\[CrossRef\]](#)
- Zhu, X.; Cao, P.; Li, P.; Yu, Y.; Guo, R.; Li, Y.; Yang, H. Bimetallic PtAu-Decorated SnO₂ Nanospheres Exhibiting Enhanced Gas Sensitivity for Ppb-Level Acetone Detection. *Nanomaterials* **2024**, *14*, 1097. [\[CrossRef\]](#)
- Bi, W.; Xiao, W.; Liu, S. Synthesis of Pt-doped SnO₂ flower-like hierarchical structure and its gas sensing properties to isopropanol. *J. Mater. Sci.* **2021**, *56*, 6095–6109. [\[CrossRef\]](#)
- Lu, S.; Zhang, Y.; Liu, J.; Li, H.-Y.; Hu, Z.; Luo, X.; Gao, N.; Zhang, B.; Jiang, J.; Zhong, A.; et al. Sensitive H₂ gas sensors based on SnO₂ nanowires. *Sens. Actuators B Chem.* **2021**, *345*, 130334. [\[CrossRef\]](#)
- Li, Z.; Tian, E.; Wang, S.; Ye, M.; Li, S.; Wang, Z.; Ma, Z.; Jiang, G.; Tang, C.; Liu, K.; et al. Single-atom catalysts: Promoters of highly sensitive and selective sensors. *Chem. Soc. Rev.* **2023**, *52*, 5088–5134. [\[CrossRef\]](#) [\[PubMed\]](#)
- Zhang, H.; Liu, G.; Shi, L.; Ye, J. Single-Atom Catalysts: Emerging Multifunctional Materials in Heterogeneous Catalysis. *Adv. Energy Mater.* **2018**, *8*, 1701343. [\[CrossRef\]](#)

15. Chen, G.; Li, R.; Huang, L. Advances in photochemical deposition for controllable synthesis of heterogeneous catalysts. *Nanoscale* **2023**, *15*, 13909–13931. [\[CrossRef\]](#)
16. Lang, R.; Du, X.; Huang, Y.; Jiang, X.; Zhang, Q.; Guo, Y.; Liu, K.; Qiao, B.; Wang, A.; Zhang, T. Single-Atom Catalysts Based on the Metal–Oxide Interaction. *Chem. Rev.* **2020**, *120*, 11986–12043. [\[CrossRef\]](#)
17. Mitchell, S.; Pérez-Ramírez, J. Atomically precise control in the design of low-nuclearity supported metal catalysts. *Nat. Rev. Mater.* **2021**, *6*, 969–985. [\[CrossRef\]](#)
18. Chu, T.; Rong, C.; Zhou, L.; Mao, X.; Zhang, B.; Xuan, F. Progress and Perspectives of Single-Atom Catalysts for Gas Sensing. *Adv. Mater.* **2022**, *35*, 2206783. [\[CrossRef\]](#)
19. Jiao, L.; Jiang, H. Metal–Organic–Framework–Based Single-Atom Catalysts for Energy Applications. *Chem* **2019**, *5*, 786–804. [\[CrossRef\]](#)
20. Liu, C.; Qian, J.; Ye, Y.; Zhou, H.; Sun, C.; Sheehan, C.; Zhang, Z.; Wan, G.; Liu, Y.; Guo, J.; et al. Oxygen evolution reaction over catalytic single-site Co in a well-defined brookite TiO₂ nanorod surface. *Nat. Catal.* **2021**, *4*, 36–45. [\[CrossRef\]](#)
21. Ye, X.; Wang, H.; Lin, Y.; Liu, X.; Cao, L.; Gu, J.; Lu, J. Insight of the stability and activity of platinum single atoms on ceria. *Nano Res.* **2019**, *12*, 1401–1409. [\[CrossRef\]](#)
22. Rong, Q.; Xiao, B.; Zeng, J.; Yu, R.; Zi, B.; Zhang, G.; Zhu, Z.; Zhang, J.; Wu, J.; Liu, Q. Pt Single Atom-Induced Activation Energy and Adsorption Enhancement for an Ultrasensitive ppb-Level Methanol Gas Sensor. *ACS Sens.* **2022**, *7*, 199–206. [\[CrossRef\]](#) [\[PubMed\]](#)
23. Sun, L.; Wang, B.; Wang, Y. High-Temperature Gas Sensor Based on Novel Pt Single Atoms@SnO₂ Nanorods@SiC Nanosheets Multi-heterojunctions. *ACS Appl. Mater. Interfaces* **2020**, *12*, 21808–21817. [\[CrossRef\]](#) [\[PubMed\]](#)
24. Koga, K. Electronic and Catalytic Effects of Single-Atom Pd Additives on the Hydrogen Sensing Properties of Co₃O₄ Nanoparticle Films. *ACS Appl. Mater. Interfaces* **2020**, *12*, 20806–20823. [\[CrossRef\]](#)
25. Yang, J.; Yan, Y.; Liang, M.; Liu, X.; Jia, R.; Ge, Y.; Li, Z.; Huang, L. Bimetallic Au–Ag-Modified ZnO-Based MEMS Sensors for ppb-Level Ethanol Detection. *IEEE Sens. J.* **2025**, *25*, 2178–2185. [\[CrossRef\]](#)
26. Liang, M.; Yan, Y.; Yang, J.; Liu, X.; Jia, R.; Ge, Y.; Li, Z.; Huang, L. In Situ-Derived N-Doped ZnO from ZIF-8 for Enhanced Ethanol Sensing in ZnO/MEMS Devices. *Molecules* **2024**, *29*, 1703. [\[CrossRef\]](#)
27. Luo, N.; Wang, C.; Zhang, D.; Guo, M.; Wang, X.; Cheng, Z.; Xu, J. Ultralow detection limit MEMS hydrogen sensor based on SnO₂ with oxygen vacancies. *Sens. Actuators B Chem.* **2022**, *354*, 130982. [\[CrossRef\]](#)
28. He, Z.; Zhang, M.; Cheng, W.; Li, X.; Zan, X.; Bao, Y.; Gu, H.; Homewood, K.; Gao, Y.; Chang, G.; et al. Reduced graphene oxide modified Nb-doped TiO₂ nanosheet for hydrogen micro-electro-mechanical system gas sensor. *Int. J. Hydrog. Energy* **2025**, *100*, 678–687. [\[CrossRef\]](#)
29. Zhang, S.; Zhao, Z.; Jia, L.; Guo, X.; Yang, R.; Deng, Q.; Zhang, D. Ultrahigh sensitive and selectivity NO₂ gas sensors based on Sn-MOF derivatives at low temperature. *Sens. Actuators B Chem.* **2024**, *417*, 136073. [\[CrossRef\]](#)
30. Xue, Z.; Yan, M.; Yu, X.; Tong, Y.; Zhou, H.; Zhao, Y.; Wang, Z.; Zhang, Y.; Xiong, C.; Yang, J.; et al. One-Dimensional Segregated Single Au Sites on Step-Rich ZnO Ladder for Ultrasensitive NO₂ Sensors. *Chem* **2020**, *6*, 3364–3373. [\[CrossRef\]](#)
31. Wang, N.; Liu, Z.; Zhou, Y.; Zhao, L.; Kou, X.; Wang, T.; Wang, Y.; Sun, P.; Lu, G. Imparting Chemiresistor with Humidity-Independent Sensitivity toward Trace-Level Formaldehyde via Substitutional Doping Platinum Single Atom. *Small* **2024**, *20*, 2310465. [\[CrossRef\]](#) [\[PubMed\]](#)
32. Xiang, T.; Yi, J. Ultrasensitive and selective hydrogen sensing of SnO₂ nanofibers decorated with Pd single atoms. *Sens. Actuators B Chem.* **2024**, *416*, 136022. [\[CrossRef\]](#)
33. Zhang, X.; Sun, J.; Tang, K.; Wang, H.; Chen, T.; Jiang, K.; Zhou, T.; Quan, H.; Guo, R. Ultralow detection limit and ultrafast response/recovery of the H₂ gas sensor based on Pd-doped rGO/ZnO-SnO₂ from hydrothermal synthesis. *Microsyst. Nanoeng.* **2022**, *8*, 67. [\[CrossRef\]](#) [\[PubMed\]](#)
34. Su, H.; Yang, H.; Ma, C.; Tang, J.; Zhu, C.; Wang, X.; Zeng, D. High Response and Selectivity of the SnO₂ Nanobox Gas Sensor for Ethyl Methyl Carbonate Leakage Detection in a Lithium-Ion battery. *ACS Sens.* **2024**, *9*, 444–454. [\[CrossRef\]](#)
35. Xiang, J.; Su, Y.; Zhang, L.; Hong, S.; Wang, Z.; Han, D.; Gu, F. Atomically Dispersed Pt on Three-Dimensional Ordered Macroporous SnO₂ for Highly Sensitive and Highly Selective Detection of Triethylamine at a Low Working Temperature. *ACS Appl. Mater. Interfaces* **2022**, *14*, 13440–13449. [\[CrossRef\]](#)
36. Xue, D.; Yuan, Y.; Yu, Y.; Xu, S.; Wei, Y.; Zhang, J.; Guo, H.; Shao, M.; Zhang, J. Spin occupancy regulation of the Pt d-orbital for a robust low-Pt catalyst towards oxygen reduction. *Nat. Commun.* **2024**, *15*, 5990. [\[CrossRef\]](#)
37. Jing, J.; Li, J.; Xue, Q.; Wang, P.; Li, W.; Meng, Y.; Zhan, Z.; Zhang, Y.; Li, F. Stable Pt/PtO₂-enhanced 3D inverse opal SnO₂ gas sensor for high sensitivity and fast H₂ sensing at low temperatures. *Sens. Actuators B Chem.* **2025**, *431*, 137462. [\[CrossRef\]](#)
38. Shekh, M.I.; Lu, T.; Kumar, A.; Sharma, G.; Zhu, G.; Stadler, F.J.; Du, B. Facile microwave-assisted synthesis of Pt single atom anchored on poly (ionic liquids) functionalized reduced graphene oxide for ultrasensitive detection of H₂O₂. *J. Environ. Chem. Eng.* **2023**, *11*, 110238. [\[CrossRef\]](#)

39. Boroun, Z.; Ghorbani, M.; Mohammadpour, R.; Moosavi, A. Origin of working temperature in H₂S sensing process of SnO₂-CuO thin bilayer: A theoretical macroscopic approach. *Sens. Actuators B Chem.* **2017**, *252*, 944–950. [[CrossRef](#)]
40. Kim, H.W.; Kwon, Y.J.; Mirzaei, A.; Kang, S.Y.; Choi, M.S.; Bang, J.H.; Kim, S.S. Synthesis of zinc oxide semiconductors-graphene nanocomposites by microwave irradiation for application to gas sensors. *Sens. Actuators B Chem.* **2017**, *249*, 590–601. [[CrossRef](#)]
41. Jang, B.; Landau, O.; Choi, S.J.; Shin, J.; Rothschild, A.; Kim, I.D. Selectivity enhancement of SnO₂ nanofiber gas sensors by functionalization with Pt nanocatalysts and manipulation of the operation temperature. *Sens. Actuators B Chem.* **2013**, *188*, 156–168. [[CrossRef](#)]
42. Zhang, S.; Chang, X.; Zhou, L.; Liu, X.; Zhang, J. Stabilizing Single-Atom Pt on Fe₂O₃ Nanosheets by Constructing Oxygen Vacancies for Ultrafast H₂ Sensing. *ACS Sens.* **2024**, *9*, 2101–2109. [[CrossRef](#)] [[PubMed](#)]
43. Kumar, S.; Lawaniya, S.D.; Agarwal, S.; Yu, Y.; Nelamarri, S.R.; Kumar, M.; Mishra, Y.K.; Awasthi, K. Optimization of Pt nanoparticles loading in ZnO for highly selective and stable hydrogen gas sensor at reduced working temperature. *Sens. Actuators B Chem.* **2023**, *375*, 132943. [[CrossRef](#)]
44. Yin, X.; Yang, Z.; Dastan, D.; Liu, Y.; Tan, X.; Gao, X.; Zhou, Y.; Li, J.; Ma, X. Sensitivity and selectivity Pt loaded SnO₂-Co₃O₄ gas sensor for hydrogen detection. *Ceram. Int.* **2023**, *49*, 38717–38725. [[CrossRef](#)]
45. Yin, X.; Guo, X. Sensitivity and selectivity of (Au, Pt, Pd)-loaded and (In, Fe)-doped SnO₂ sensors for H₂ and CO detection. *J. Mater. Sci. Mater. Electron.* **2014**, *25*, 4960–4966. [[CrossRef](#)]
46. Li, G.; Shen, Y.; Zhao, S.; Li, A.; Gao, S.; Wei, D.; Yuan, Z.; Meng, F.; Meng, D. High response and moisture resistance hydrogen sensors based on sandwich-structured PtSnx-rGO-SnO₂ nanocomposites. *Sens. Actuators B Chem.* **2022**, *368*, 132146. [[CrossRef](#)]
47. Barsan, N.; Weimar, U. Conduction Model of Metal Oxide Gas Sensors. *J. Electroceramics* **2001**, *7*, 143–167. [[CrossRef](#)]
48. Gurlo, A.; Riedel, R. In Situ and Operando Spectroscopy for Assessing Mechanisms of Gas Sensing. *Angew. Chem. Int. Ed.* **2007**, *46*, 3826–3848. [[CrossRef](#)]
49. Yu, F.; Lang, Z.; Yin, L.; Feng, K.; Xia, Y.; Tan, H.; Zhu, H.; Zhong, J.; Kang, Z.; Li, Y. Pt-O bond as an active site superior to Pt⁰ in hydrogen evolution reaction. *Nat. Commun.* **2020**, *11*, 490. [[CrossRef](#)]
50. Murata, N.; Suzuki, T.; Lin, Y.; Nitani, H.; Niwa, Y.; Wada, T.; Uo, M.; Asakura, K. Structure of Atomically Dispersed Pt in a SnO₂ Thin Film under Reaction Conditions: Origin of Its High Performance in Micro Electromechanical System Gas Sensor Catalysis. *ACS Appl. Mater. Interfaces* **2022**, *14*, 39507–39514. [[CrossRef](#)]
51. Rao, C.; Shen, J.; Wang, F.; Peng, H.; Xu, X.; Zhan, H.; Fang, X.; Liu, J.; Liu, W.; Wang, X. SnO₂ promoted by alkali metal oxides for soot combustion: The effects of surface oxygen mobility and abundance on the activity. *Appl. Surf. Sci.* **2018**, *435*, 406–414. [[CrossRef](#)]
52. Yang, L.; Zhou, X.; Song, L.; Wang, Y.; Wu, X.; Han, N.; Chen, Y. Noble Metal/Tin Dioxide Hierarchical Hollow Spheres for Low-Concentration Breath Methane Sensing. *ACS Appl. Nano Mater.* **2018**, *1*, 6327–6336. [[CrossRef](#)]
53. Wang, D.; Xu, M.; Li, K.; Yan, Y.; Chen, X.; Yu, D.; Jia, R.; Huang, L. Enhanced ethanol detection: A high-performance MEMS sensor utilizing Pt-modified SnO₂-TiO₂ composite nanomaterials. *Surf. Interfaces* **2025**, *68*, 106679. [[CrossRef](#)]
54. Pylnev, M.; Nishikubo, R.; Ishiwari, F.; Wakamiya, A.; Saeki, A. Sequential Deposition of Diluted Aqueous SnO₂ Dispersion for Perovskite Solar Cells. *Sol. RRL* **2024**, *8*, 2400415. [[CrossRef](#)]
55. Hai, X.; Xi, S.; Mitchell, S.; Harrath, K.; Xu, H.; Akl, D.F.; Kong, D.; Li, J.; Li, Z.; Sun, T.; et al. Scalable two-step annealing method for preparing ultra-high-density single-atom catalyst libraries. *Nat. Nanotechnol.* **2021**, *17*, 174–181. [[CrossRef](#)]
56. Yan, Y.; Lan, X.; Li, Y.; Liang, M.; Liu, X.; Yang, J.; Jia, R.; Ge, Y.; Li, Z.; Huang, L. Oxygen-Vacancy-Rich SnO₂ Nanoparticles Based Ultralow-Power MEMS Sensor for Nitrogen Dioxide Detection. *ACS Appl. Nano Mater.* **2025**, *8*, 6920–6929. [[CrossRef](#)]
57. Zhou, Q.; Xu, L.; Umar, A.; Chen, W.; Kumar, R. Pt nanoparticles decorated SnO₂ nanoneedles for efficient CO gas sensing applications. *Sens. Actuators B Chem.* **2018**, *256*, 656–664. [[CrossRef](#)]

Disclaimer/Publisher's Note: The statements, opinions and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and/or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions or products referred to in the content.