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Enhanced Hydrogen Sensing Using Palladium-Functionalized MoO3-x Nanosheets Synthesized via Anodic Oxidation

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Summary:

Defective metal oxides are promising for gas sensing due to enhanced free carrier concentrations. In this work, reduced molybdenum oxide nanosheets (MoO_{3-x} NSs) were synthesized on alumina substrates using a fast electrochemical method and decorated with palladium chloride. Structural and morphological analysis confirmed temperature-dependent properties. Hydrogen sensing tests showed that operating temperature significantly affects response and recovery times, demonstrating the material's potential for efficient gas sensor applications.

Keywords: hydrogen sensing, reduced MoO3 nanosheets, Metal oxide, Palladium, Response time.

Background

Hydrogen, a clean and efficient energy carrier, is used in fuel cells, electric vehicles, and power systems. However, its wide flammability range (4–75%), low ignition energy, and invisibility pose significant safety risks, making hydrogen sensors crucial for early leak detection in industrial settings at low concentrations [1,2]. Effective sensors must offer high sensitivity and selectivity under real-world conditions. Resistive sensors based on semiconductor metal oxides are preferred due to their stability, simplicity, and scalability. Molybdenum trioxide (MoO_3), with its unique layered structure and high electron mobility, shows promise, especially in its substoichiometric form (MoO_{3-x}), which exhibits enhanced conductivity and gas-sensing performance due to oxygen vacancies [3]. This study focuses on synthesizing MoO_{3-x} nanosheets via anodic oxidation and functionalizing them with $PdCl_2$ to improve hydrogen sensitivity, aiming to develop a cost-effective, fast-response sensor.

Description of the New Method and system

MoO_{3-x} nanosheets were synthesized through anodic oxidation of high-purity molybdenum in 0.02 M HCl at 30 V for 15 minutes, yielding oxygen vacancy-rich structures with enhanced conductivity. The nanosheets were drop-cast onto Al_2O_3 substrates with gold electrodes and functionalized with $PdCl_2$ to boost hydrogen adsorption. Hydrogen sensing was evaluated in a custom chamber using static H_2 injection (10% H_2/Ar, diluted), alternating 210 s exposure and 180 s recovery under vacuum at 150–300 °C. Ar served as the carrier gas, ambient air enabled recovery, and RH was kept at ~30%. Electrical signals were recorded via IVIUMSTAT potentiostat, with sensor response calculated as:

$$\mathrm{Re}sponce = \frac{R_{air} - R_{gas}}{R_{gas}} \qquad (1)$$

Where R_{air} and R_{gas} are the resistances in air and hydrogen, respectively.

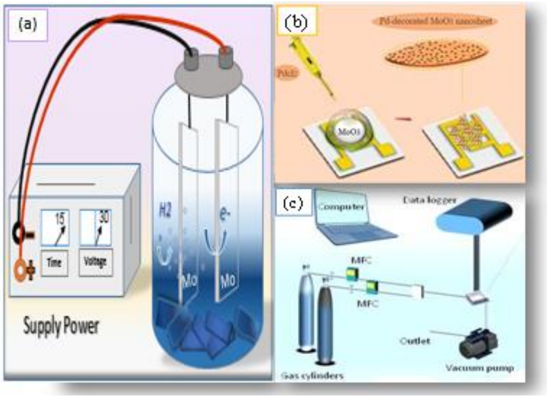

Figure 1 Schematic of (a) anodic synthesis of MoO_{3-x} nanosheets, (b) Pd functionalization on gold electrodes, and (c) hydrogen gas sensing system.

Results and Discussion

The structural and morphological properties of MoO_{3-x} nanosheets (NSs) were analyzed using XRD, Raman spectroscopy, FESEM, and TEM. XRD (Fig. 2a) revealed a nanocrystalline structure with broad peaks, indicating partial disorder.

Raman spectroscopy (Fig. 2b) identified characteristic O–Mo–O and Mo=O vibrations, suggesting oxygen vacancies. FESEM (Fig. 2c) showed a wrinkled, layered structure, while TEM (Fig. 2d) confirmed stacked nanosheets with dispersed nanoparticles, enhancing the surface area for gas sensing. XPS analysis (inset of Fig. 2d) confirmed the presence of molybdenum, oxygen, and residual carbon.

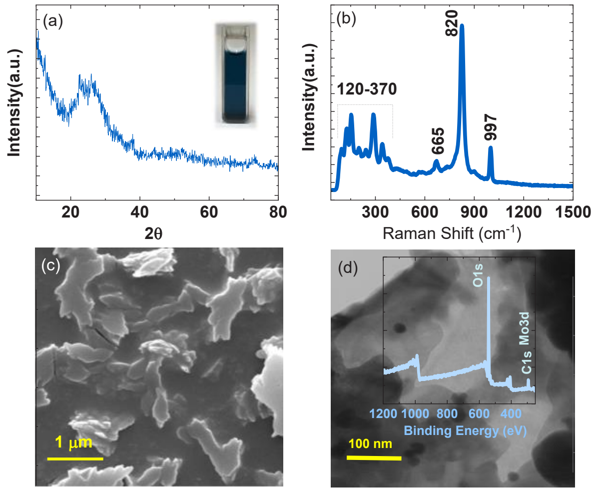

Figure 2 *MoO_{3-x} NSs charecterization, (a) XRD, (b) Raman spectroscopy, (c) SEM, (d) TEM(inside: XPS survey scan).*

Hydrogen sensing performance of the MoO_{3-x} NSs was assessed by monitoring the variation in electrical resistance under controlled gas exposure at different operating temperatures (Fig. 3a). When 10% H_2 gas was injected for 210 seconds, a notable increase in electrical current (decrease in resistance) was observed, especially at elevated temperatures. This behavior aligns with the n-type semiconducting nature of molybdenum oxide, where hydrogen exposure donates electrons and enhances conductivity. As shown in Fig. 3b, the sensor response significantly increased with temperature, peaking at 300 °C. The response was reversible upon returning to air, demonstrating excellent repeatability and stability. Figure 3c highlights the response after 210 seconds of hydrogen exposure, with negligible sensitivity at 150 °C and sharp increases above 250 °C, reaching maximum values exceeding 22200(222/%, in diagram). This temperature dependence emphasizes the importance of high temperatures for sensor performance. Figure 3d shows the response and recovery times, with response times under 210 seconds and recovery times under 8 seconds, demonstrating fast kinetics. This combination of high response, rapid recovery, and stability makes Pd-decorated MoO_{3-x} NSs promising for efficient, low-cost hydrogen sensing, particularly in high-temperature environments.

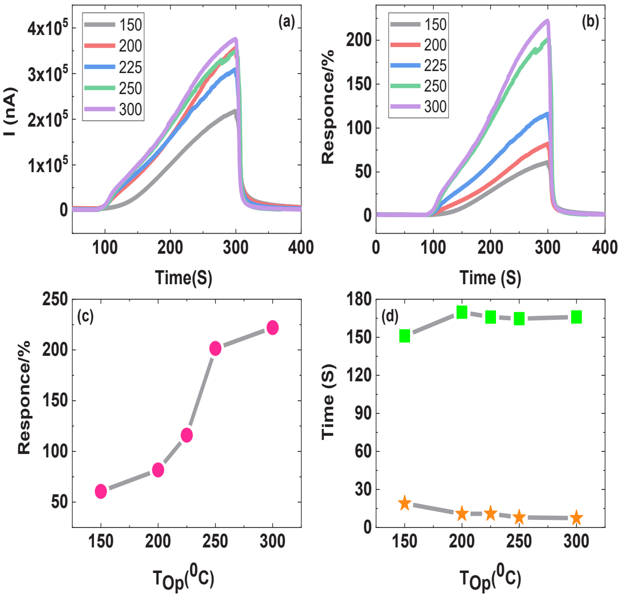

Figure 3 *(a) Current variation of MoO_{3-x} NSs during H_2 exposure; (b) dynamic response of Pd-functionalized NSs at different temperatures; (c) response at 210 s; (d) response/recovery times vs. temperature.*

The MoO_3 NSs demonstrated high selectivity for hydrogen at 300 °C, showing significantly higher response to H_2 than to CO_2, NO_2, CH_4, and ethanol, confirming their specificity and suitability for reliable gas sensing applications.

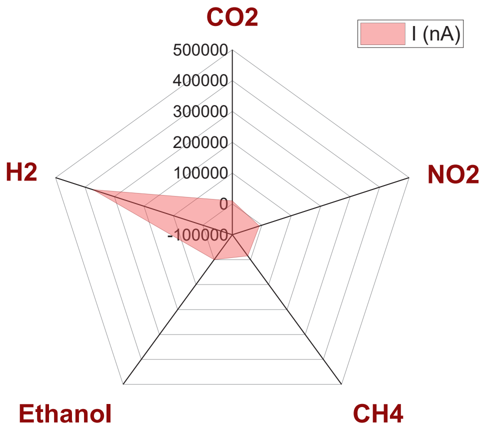

Figure 4 *Hydrogen selectivity of sample at the operation temperature of 300 °C.*

References

[1] M. Yue, H. Lambert, E. Pahon, R. Roche, S. Jemei, D. Hissel, Hydrogen energy systems: A critical review of technologies, applications, trends and challenges, Renew. Sustain. Energy Rev. 146 (2021).

[2] S.H. Kim, K.S. Yun, Room-temperature hydrogen gas sensor composed of palladium thin film deposited on NiCo2O4 nanoneedle forest, Sensors Actuators B Chem. 376 (2023).

[3] Q. Li, L. Wang, A. Xiao, L. Zhu, Z. Yang, Hydrogen sensing towards palladium-based nanocomposites: A review, Int. J. Hydrogen Energy. (2024).