

Pd Decorated WS₂ Synthesized via Facile APCVD Technique for Highly Selective Room Temperature H₂ sensing

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Summary:

Developing sensitive hydrogen gas sensors is crucial for detecting and managing hazards in storage, transport, and leakages. While metal oxide gas sensors (MOX) suffer from poor selectivity and high temperatures, attention has shifted to two-dimensional materials like tungsten disulfide (WS₂). However, bare WS₂ sensors face limitations like slow response-recovery times and low sensitivity. Functionalizing TMDs with noble metal nanoparticles, notably Pd, enhances sensor performance. This study presents a highly sensitive Pd-decorated WS₂ composite based sensor for room temperature (RT) H₂ sensing, with a remarkable response of 55% (Pd quantity (3 mg)).

Keywords: Gas sensor, TMDs, hydrogen, APCVD, nanomaterials

Background, Motivation and Objective

The growing demand for sustainable energy sources has spotlighted hydrogen (H₂) as a leading contender for the next generation of energy, offering the potential to replace fossil fuels and significantly reduce greenhouse gas emissions [1]. However, its high explosiveness and flammability necessitate careful storage and handling to mitigate safety risks. Developing sensitive and selective hydrogen gas sensors is crucial for detecting and managing potential hazards associated with hydrogen storage, transport, and leakage. While metal oxide gas sensors (MOX) have been widely used, they suffer from limitations such as poor selectivity and high operating temperatures. Recently, attention has turned to two-dimensional materials (2D) like graphene and transition metal dichalcogenides (TMDs) such as tungsten disulfide (WS₂) for improved sensing capabilities [2]. WS₂ has garnered significant interest due to its exceptional properties. Various research works have demonstrated the potential of WS₂-based sensors for detecting gases including hydrogen. However, bare WS₂ sensors face challenges like slow response-recovery speeds and low sensitivity. Functionalizing the host matrix with noble metal nanoparticles especially Pd enhances sensor performance through electronic and chemical sensitization mechanisms [1]. Optimization of nanoparticle decoration is crucial to ensure sensor efficacy. In this study, a highly sensitive Pd-decorated WS₂ composite for room temperature H₂ sensing demonstrated

superior performance, with highest Pd quantity (3 mg) exhibiting the highest response of 55%. The amount of ethanol and WS₂ was kept constant at 10 ml and 10 mg respectively in all the solutions.

Materials and Methods

WS₂ was synthesized as reported in our earlier study [2]. To decorate the WS₂ with Pd, commercial Pd powder was mixed with WS₂ in varying proportions (0.5 mg, 1 mg and 3 mg) and subjected to 90 minutes of sonication in absolute ethanol. The resulting solutions were airbrushed onto gold interdigitated electrodes on silicon substrates fabricated using laser lithography.

Results

Fig. 1 (a and b) illustrate morphology of WS₂ and Pd- WS₂ composite respectively. As seen from the Field Emission Scanning Electron Microscopy (FESEM) images, the deposited WS₂ depicts flake like morphology with an average size ranging from 100 nm to 1.2 μm. Also, the Pd nanoparticles can be seen in the form of clusters (in red circles) with an average particle size of 50 nm (Fig. 1 (b)). The Pd clusters are uniformly distributed all over the WS₂ flakes. The samples were further characterized using transmission Electron Microscopy (TEM). Color mapping of the samples show successful decoration of WS₂ with Pd nanoparticles as shown in Fig. 2 (a). Fig. 2 shows EDX spectrum of the Pd-WS₂ composite.

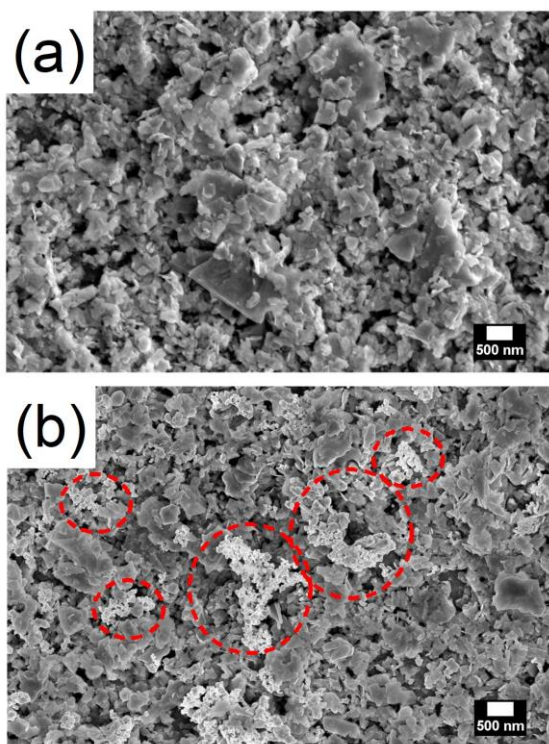


Fig. 1. FSEM images of (a) WS_2 , (b) Pd decorated WS_2 (Red circles indicate Pd clusters).

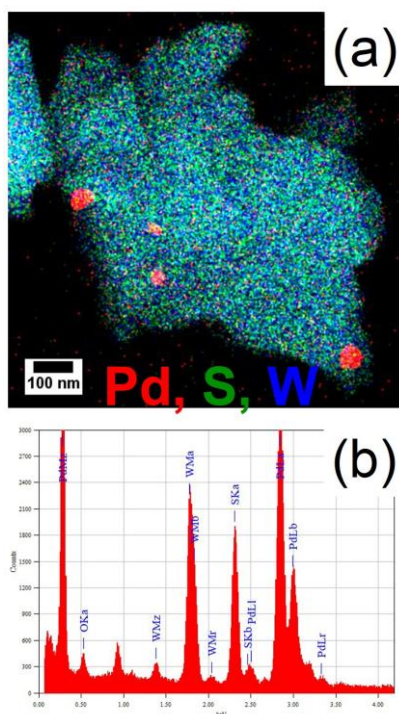


Fig. 2. (a) Color mapping of Pd decorated WS_2 nanocomposite, (b) EDS spectrum of Pd decorated WS_2 nanocomposite.

We tested bare WS_2 and Pd-decorated WS_2 sensors with varying amounts of Pd (0.5 mg, 1 mg, and 3 mg) towards 500 ppm of H_2 at room temperature (RT), $100^\circ C$, and $150^\circ C$. The sensor with 3 mg of Pd exhibited the highest sensitivity towards 500 ppb of H_2 at RT. Fig. 3 (a)

illustrates the sensor resistance dynamics towards 500 ppm of H_2 at RT. The pristine WS_2 sensor showed no response to any concentrations of H_2 but began to respond with small responses at elevated temperatures of $150^\circ C$, still lower than the responses shown by the best sensor at RT. The sensor responses are stable and repeatable. Fig. 3 (b) demonstrates that the sensing response decreases almost linearly with increasing operating temperature. Gas sensing response of the best responding sensor are presented. The detailed selectivity and humidity sensing responses will be presented in the conference.

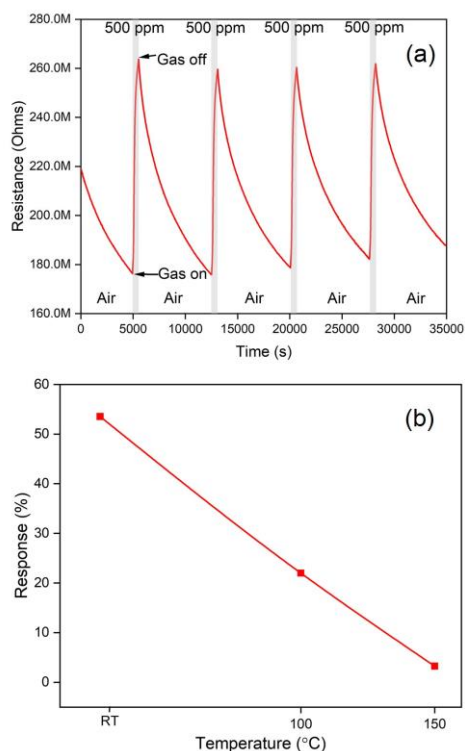


Fig. 3. (a) Resistance dynamics of 3 mg Pd decorated WS_2 , (b) the sensor response as a function of temperature towards 500 ppm H_2 at RT.

Conclusions

This study presents a highly sensitive Pd-decorated WS_2 composite for room temperature H_2 sensing, achieving a remarkable 55% response with 3 mg of Pd.

References

- [1] S. B. Malik, F. E. Annanouch, and E. Llobet, "Pd-Nanoparticle-Decorated Multilayered MoS_2 Sheets for Highly Sensitive Hydrogen Sensing," *Chemosensors*, vol. 11, no. 11, p. 550, Oct. 2023, doi: 10.3390/chemosensors11110550.
- [2] S. B. Malik, F. E. Annanouch, and E. Llobet, "Room Temperature Detection of Ppb Level NO_2 by WS_2 Sensors," *Proc. IEEE Sensors*, no. 2, pp. 1–4, 2023, doi: 10.1109/SENSOR56945.2023.10324906.