

CuO-decorated SnO₂ Nanowires Modified Femtosecond Laser for H₂S Detection

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ABSTRACT: Femtosecond laser (fs-laser) processing has emerged as a powerful tool for tailoring surface properties of metal oxides, yet its application in gas sensing remains relatively unexplored. In this work, fs-laser irradiation was employed to engineer the surface of CuO-decorated SnO₂ nanowires for improved H₂S gas sensing. Structural and morphological analyses confirmed that the nanowire framework was preserved after fs-laser treatment, while X-ray photoelectron spectroscopy revealed noticeable modifications in surface oxygen species and defect-related states. The formation of CuO on the SnO₂ surface enabled the creation of CuO–SnO₂ p–n heterojunctions, which play a key role in modulating the charge transport behavior. Gas sensing measurements toward H₂S demonstrated that the fs-laser treated CuO-decorated SnO₂ NWs exhibited higher responses compared to untreated counterparts at 150°C, the optimal operating temperature. The response to 10 ppm H₂S increased from 1.12 for pristine SnO₂ NWs to 1.42 for Cu-decorated SnO₂ NWs, 1.72 for fs-laser treated SnO₂ NWs, and 5.38 for the fs-laser treated CuO-decorated SnO₂ NWs. The corresponding response and recovery time toward 10 ppm H₂S at this temperature was 462 s and 850 s, respectively. The enhanced sensing performance is attributed to the combined effects of fs-laser-induced surface defects and the heterojunctions between CuO and SnO₂. Furthermore, the well-known chemical interaction between CuO and H₂S, involving the conversion of CuO to CuS, is considered to contribute to the pronounced resistance modulation. This study highlights that fs-laser surface engineering provides a simple and effective route to improve the H₂S sensing characteristics of metal oxide nanowires, offering insights into defect-mediated gas sensing mechanisms and practical sensor design.

KEYWORDS: SnO₂ nanowires, CuO nanostructure, Gas sensor, Heterojunction, H₂S detection

1. INTRODUCTION

Hydrogen sulfide (H₂S) is a hazardous and corrosive gas frequently encountered in petroleum refining, wastewater treatment, and chemical industries [1,2]. Due to its toxicity and potential to cause severe health and safety issues, reliable detection of H₂S is essential for environmental monitoring and

industrial safety. Chemiresistive gas sensors based on semiconductor metal oxides have been widely adopted for this purpose because of their simplicity, low fabrication cost, and compatibility with miniaturized devices [3,4]. However, achieving high sensitivity and selectivity toward H₂S still requires precise control over the surface and interfacial properties of sensing materials.

While extensive research has focused on developing new sensing materials, increasing attention is now being directed toward process-based approaches that modify surface states without fundamentally altering the material system. Such approaches are attractive because they can enhance sensing performance while maintaining the advantages of well-established materials. In chemiresistive sensors, surface defects, oxygen-related species, and interfacial charge distribution critically influence gas adsorption and electron

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transfer processes [5-7]. Therefore, engineering these surface features through controllable processing methods represents a promising route to improve sensing characteristics.

Femtosecond (fs) laser processing has emerged as a powerful tool for surface engineering due to its ultrashort pulse duration and highly localized energy deposition [8-10]. Unlike conventional thermal or chemical treatments, fs-laser irradiation can induce non-equilibrium structural and chemical modifications with minimal heat-affected zones. This enables precise tuning of surface morphology, defect states, and chemical composition. Fs-laser techniques have been successfully applied in micro/nanostructuring, surface texturing, and functional modification of various materials [11-13]. Nevertheless, their use as a post-treatment strategy for gas sensing materials remains relatively underexplored.

In the context of gas sensing, modifying surface defects and hetero-interfaces is particularly important because these factors govern adsorption-desorption kinetics and resistance modulation. SnO₂ NWs represent a well-established sensing platform with stable baseline characteristics, making them suitable for process-oriented studies. Furthermore, the incorporation of copper oxide species on SnO₂ is known to introduce p-n heterojunctions and strong chemical interactions with H₂S [14,15]. However, most previous studies have emphasized compositional modification rather than process-induced surface engineering.

In this work, we focus on femtosecond laser irradiation as a surface engineering process to tailor the sensing properties of CuO-decorated SnO₂ NWs. Rather than developing a new material system, this study investigates how fs-laser processing alters surface states and defect chemistry, and how these changes influence H₂S sensing behavior. By comparing untreated and fs-laser treated samples, the role of process-induced surface modification is systematically evaluated. This process-centered approach provides insights into how advanced laser processing can be utilized to tune gas sensing performance without complex material redesign, offering a practical pathway for sensor optimization.

2. EXPERIMENTAL

2.1 Materials

High-purity tin (Sn) powder ($\geq 99.9\%$) was used as the source material for nanowire growth. Silicon (Si) substrates were employed as growth substrates, and a thin gold (Au) layer served as the catalyst for vapor-liquid-solid (VLS) growth. Argon (Ar, $\geq 99.999\%$) and oxygen (O₂, $\geq 99.999\%$) gases were used as carrier and reactive gases, respectively, during the growth process. All chemicals and gases were of

analytical grade and used as received without further purification. Deionized (DI) water and ethanol were used for substrate cleaning prior to catalyst deposition.

A high-purity copper (Cu) target ($\geq 99.99\%$) was used as the source material for the deposition of the copper component, which was introduced to form the catalytic p-type CuO species responsible for heterojunction formation and H₂S chemical sensitization.

2.2 Synthesis of SnO₂ NWs

SnO₂ NWs were synthesized using a catalyst-assisted vapor-liquid-solid (VLS) growth process in a horizontal tube furnace. Prior to growth, Si substrates were sequentially cleaned with acetone, ethanol, and DI water under ultrasonication and then dried under a nitrogen stream. A thin Au catalyst layer was deposited onto the cleaned Si substrates by sputtering.

For nanowire growth, high-purity Sn powder was placed in an alumina boat and positioned at the center of the quartz tube, while the Au-coated Si substrates were located downstream. The system was purged with inert gas to remove residual air before heating. The furnace was then heated to the growth temperature under a controlled Ar/O₂ gas flow. During heating, Sn vapor was generated and transported to the substrate region, where Au-Sn liquid alloy droplets formed and served as catalytic sites for nanowire growth. In the presence of oxygen, Sn vapor reacted to form SnO₂, leading to the nucleation and elongation of SnO₂ NWs via the VLS mechanism.

After growth, the furnace was naturally cooled to room temperature under inert gas flow. The as-grown SnO₂ NWs were directly used for subsequent surface modification and gas sensing experiments without further treatment. The synthesized material was denoted as pristine SnO₂ NWs.

2.3. Formation of Cu on the SnO₂ NWs

Cu was sputtered onto the synthesized SnO₂ NWs using a DC sputtering system operated at 10 mA for 1 min, corresponding to a nominal thickness of 3 nm. The resulting samples were denoted as Cu-decorated SnO₂ NWs.

2.4. Femtosecond Laser Irradiation

Femtosecond laser irradiation was employed as a post-treatment to modify the surface states of the CuO-decorated SnO₂ nanowires. The laser system operated at a wavelength of 1030 nm, with a pulse duration of 33.6 fs, a repetition rate of 5 kHz, and a pulse energy of 1.2 mJ. The focused laser beam was scanned across the sample surface at a scanning speed of

1.5 mm s⁻¹ using a galvanometer scanner to ensure uniform irradiation. The pulse energy was controlled using a half-wave plate and a polarizing beam splitter. All laser-treated specimens were processed in the same batch using an identical fixed scan program.

A galvanometer scanner was used to scan the focused laser beam across the sample surface, enabling uniform irradiation. The ultrashort pulse duration allowed localized energy deposition with minimal thermal diffusion, thereby inducing surface defect formation and modification of oxygen-related species without damaging the nanowire framework.

Such fs-laser treatment is known to introduce defect sites and alter surface chemistry, which can enhance gas adsorption and charge transfer during sensing. The resulting samples were denoted as fs-laser treated CuO-decorated SnO₂ NWs, while the pristine SnO₂ NWs subjected to the same fs-laser treatment were denoted as fs-laser treated SnO₂ NWs.

2.5. Materials characterization

The morphologies of the synthesized nanostructures were characterized using field-emission scanning electron microscopy (FE-SEM; Regulus 8220, Hitachi Ltd., Japan) and transmission electron microscopy (TEM; Talos F200X, Thermo Fisher Scientific, USA). Elemental compositions and spatial distributions were examined by energy-dispersive X-ray spectroscopy (EDS) coupled with high-angle annular dark-field scanning TEM (HAADF-STEM). The crystalline phases and structural properties of the samples were analyzed using X-ray diffraction (XRD; Empyrean, PANalytical, Netherlands). Surface chemical states and elemental valence information were investigated by X-ray photoelectron spectroscopy (XPS; NEXSA, Thermo Fisher Scientific, USA).

2.6. Gas sensing measurements

The synthesized nanostructures were dispersed in deionized water and drop-cast onto alumina substrates patterned with Au interdigitated electrodes. After drying under ambient conditions, the devices were installed in a sealed test chamber for gas sensing measurements. Target gases were introduced by diluting certified high-purity gases with synthetic air using mass flow controllers, and the total gas flow rate was maintained at 500 sccm throughout the measurements.

The electrical resistance of the sensors was continuously monitored in air (R_a) and in the presence of target gases (R_g). The sensor response was defined as R_a/R_g for reducing gases and R_g/R_a for oxidizing gases. Each sensing cycle consisted of 500 s exposure to the target gas followed by 1000 s recovery in air. The response time and recovery time were defined as the

time required for the resistance to reach 90% of the total change upon gas exposure and to return to 90% of the baseline resistance after gas removal, respectively.

3. RESULTS AND DISCUSSIONS

3.1 Morphological and chemical studies

Fig. 1 shows the morphological and compositional characteristics of the SnO₂ NWs before and after Cu decoration and fs laser irradiation. The pristine SnO₂ NWs (Fig. 1(a)) exhibit a typical one-dimensional morphology with smooth surfaces and uniform diameters. After Cu decoration (Fig. 1(b)), the overall nanowire structure is preserved, while small nanoparticles are observed on the nanowire surface, indicating successful decoration of Cu-based species without

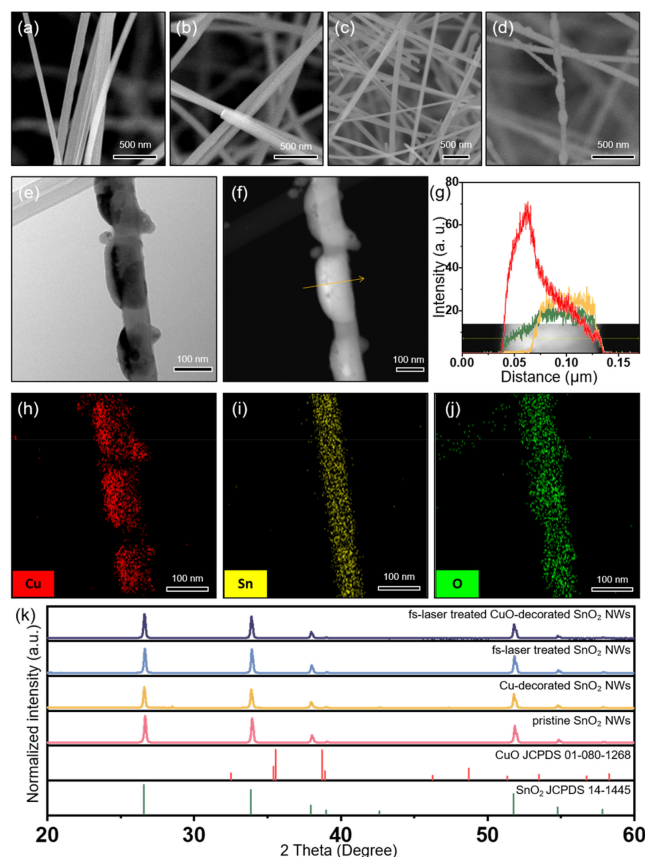


Fig. 1. Structural characterization of SnO₂-based NWs. (a-d) SEM images of (a) pristine SnO₂ NWs, (b) Cu-decorated SnO₂ NWs, (c) fs-laser treated SnO₂ NWs, (d) fs-laser treated CuO-decorated SnO₂ NWs. (e) TEM image of fs-laser treated CuO-decorated SnO₂ NWs. (f, g) HAADF-STEM image and EDS line profile of fs-laser treated CuO-decorated SnO₂ NWs. (h-j) TEM-EDS mapping of (h) Cu, (i) Sn, (j) O in fs-laser treated CuO-decorated SnO₂ NWs. (k) XRD spectra of pristine SnO₂ NWs, Cu-decorated SnO₂ NWs, fs-laser treated SnO₂ NWs, and fs-laser treated CuO-decorated SnO₂ NWs.

altering the NW framework.

Following fs-laser irradiation (Fig. 1(c,d)), the nanowire morphology remains largely intact, suggesting that the ultrashort laser pulses did not induce significant thermal damage or melting of the structures. However, a slightly roughened surface texture can be discerned, which is commonly associated with localized surface modification induced by fs-laser processing. Such surface perturbations are known to influence defect formation and surface reactivity.

The TEM image of the fs-laser treated CuO-decorated SnO₂ NWs sample (Fig. 1(e)) confirms the crystalline nanowire structure and the presence of surface-attached nanoparticles. The HAADF-STEM image (Fig. 1(f)) further reveals clear contrast between the nanowire body and the decorated particles due to Z-contrast. The line profile (Fig. 1(g)) and elemental mapping (Fig. 1(h–j)) demonstrate that Sn and O are uniformly distributed along the nanowire, while Cu is localized on the surface, verifying successful surface decoration rather than bulk incorporation.

X-ray diffraction patterns of the samples are presented in Fig. 1(k). All major diffraction peaks can be indexed to the tetragonal rutile structure of SnO₂ [16], indicating that the crystalline phase of SnO₂ is well preserved after Cu decoration and femtosecond laser treatment. No noticeable peak shift or broadening is observed, suggesting that the bulk crystal structure of SnO₂ remains stable during the surface modification processes.

Distinct diffraction peaks corresponding to CuO are not clearly detected. This can be attributed to the low loading amount and high dispersion of Cu-based species on the nanowire surface, which may fall below the detection limit of conventional XRD. Similar observations have been reported in previous studies where surface-decorated metal oxides with low content or nanoscale dispersion were not readily detectable by XRD [17].

Nevertheless, the presence of CuO species is supported by XPS analysis, which reveals characteristic Cu²⁺ features and satellite peaks. Therefore, the combined XRD and XPS results suggest that CuO is present primarily as highly dispersed surface species rather than as a bulk crystalline phase.

The surface chemical states were further analyzed by XPS (Fig. 2). The survey spectra confirm the presence of Sn, O, and Cu elements. The Sn 3d spectra (Fig. 2(b,c)) display two characteristic peaks corresponding to Sn⁴⁺, consistent with the SnO₂ phase. The Sn 3d peaks of the fs-laser treated CuO-decorated SnO₂ NWs are shifted toward lower binding energy, indicating modified surface electronic states of SnO₂ induced by the interaction with CuO.

Meanwhile, the O 1s spectra can be deconvoluted into lattice oxygen (O²⁻), defect-related oxygen, and surface hydroxyl

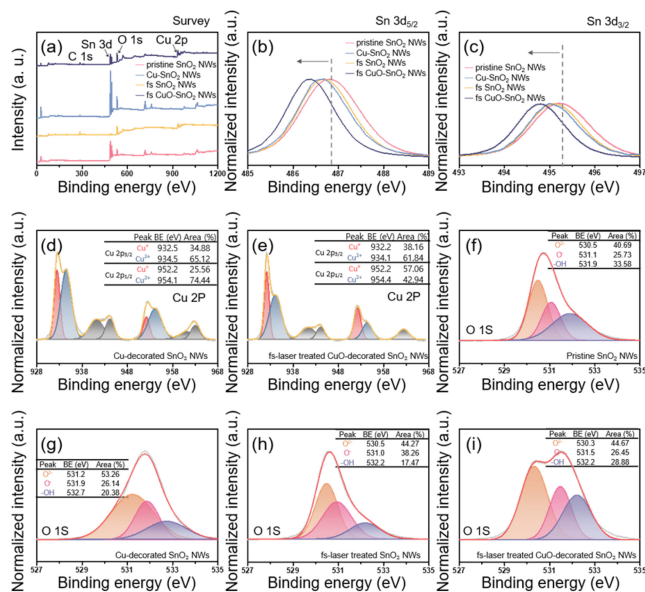


Fig. 2. XPS analysis of pristine SnO₂ NWs, Cu-decorated SnO₂ NWs, fs-laser treated SnO₂ NWs, and fs-laser treated CuO-decorated SnO₂ NWs. (a) Survey spectra of all samples. (b, c) Sn 3d, (d, e) Cu 2p, and (f–i) O 1s.

species [18]. The relative fraction of defect-related oxygen shows variations after fs-laser irradiation, suggesting that the laser treatment modifies the surface defect chemistry. Such defect states are generally considered active sites for gas adsorption and charge transfer, and therefore can influence sensing behavior [19].

The Cu 2p spectra exhibit features characteristic of oxidized copper species, including satellite peaks associated with Cu²⁺, indicating that Cu is predominantly present as CuO on the SnO₂ surface. The formation of CuO–SnO₂ interfaces is important for establishing p–n heterojunctions, which can modulate the depletion layer and charge transport properties of the sensor [20,21].

Overall, the morphological and chemical analyses indicate that fs-laser irradiation primarily modifies the surface states without destroying the nanowire architecture, while Cu decoration introduces catalytically active CuO species. These features are expected to collectively influence the H₂S sensing performance discussed in the following section.

3.2. Gas sensing studies

The H₂S sensing performances of the sensors based on pristine SnO₂ NWs, Cu-decorated SnO₂ NWs, fs-laser treated SnO₂ NWs, and fs-laser treated CuO-decorated SnO₂ NWs were systematically evaluated. Fig. 3(a,d,g,j) present the dynamic responses toward 10 ppm H₂S at different operating temperatures of 100, 150, 200, and 300°C, respectively. All

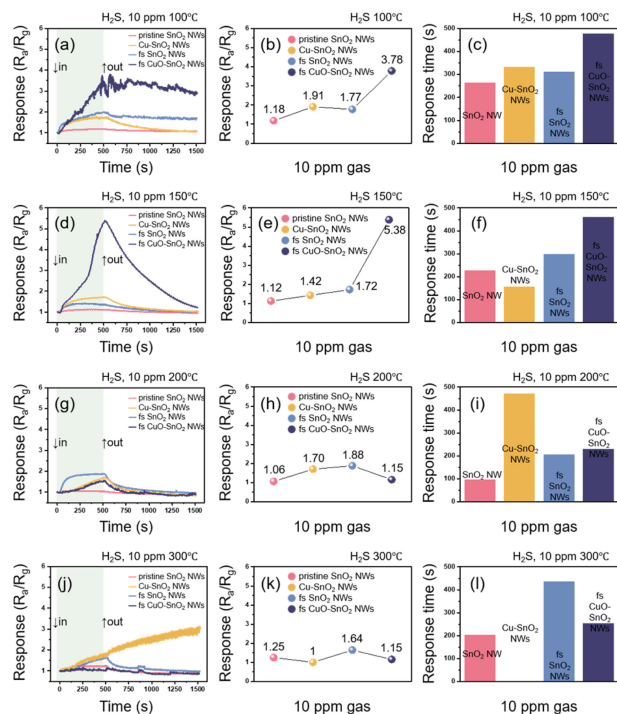


Fig. 3. Gas-sensing performance of SnO₂ NW-based sensors toward 10 ppm H₂S at different operating temperatures. (a–c) Dynamic response curves, response values, and response and recovery times at 100°C; (d–f) 150°C; (g–i) 200°C; and (j–l) 300°C.

sensors exhibit a typical decrease in resistance upon exposure to H₂S, confirming their n-type sensing behavior toward reducing gases.

Among the tested devices, the fs-laser treated CuO-decorated SnO₂ NWs sensor shows the highest response at 150°C, which is identified as the optimal operating temperature.

The enhanced response at this temperature suggests a balanced condition between adsorption and desorption kinetics of H₂S molecules. At lower temperatures, insufficient activation of surface reactions likely limits the response, whereas at higher temperatures, accelerated desorption reduces gas–surface interaction time.

Fig. 3(b,e,h,k) present the responses of the four sensors toward 10 ppm H₂S at 100, 150, 200, and 300°C, respectively, and the corresponding response and recovery times are summarized in Fig. 3(c,f,i,l). At the optimal operating temperature of 150°C, the fs-laser treated CuO-decorated SnO₂ NWs sensor exhibits a response of 5.38, with response and recovery times of approximately 462 s and 850 s, respectively.

The concentration-dependent responses measured at 150°C are shown in Fig. 4(a–e) and Fig. 5. The response increases monotonically with H₂S concentration from 2 to 10 ppm, demonstrating reliable concentration-dependent behavior.

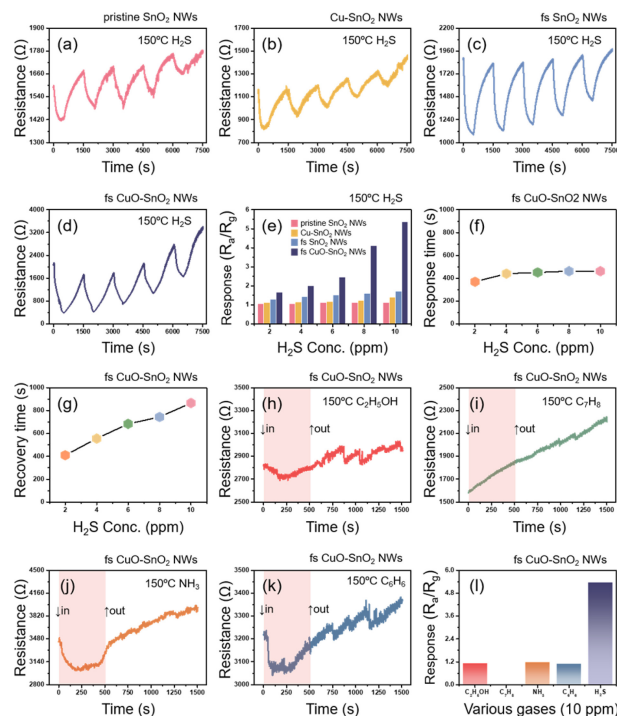


Fig. 4. Gas-sensing performance of SnO₂ NW-based sensors at 150°C. (a–d) Dynamic resistance responses of pristine SnO₂ NWs, Cu-decorated SnO₂ NWs, fs-laser treated SnO₂ NWs, and fs-laser treated CuO-decorated SnO₂ NWs sensors toward various H₂S concentrations, respectively. (e) Variation in the responses of the sensors with increasing H₂S concentration. (f, g) Response and recovery times of the fs-laser treated CuO-decorated SnO₂ NWs sensor as a function of H₂S concentration. (h–k) Dynamic responses of the fs-laser treated CuO-decorated SnO₂ NWs sensor toward 10 ppm ethanol, toluene, ammonia, and benzene, respectively. (l) Selectivity toward various gases at 150°C.

Even at low concentrations, measurable responses are observed, indicating good sensitivity.

Selectivity is a critical factor for practical gas sensors. As shown in Fig. 4(h–l), the fs-laser treated CuO-decorated SnO₂ NWs sensor exhibits a significantly higher response to H₂S compared to other interfering gases such as 10 ppm ethanol, toluene, ammonia, and benzene. This selective behavior is commonly associated with the strong chemical affinity between CuO and H₂S.

The improved H₂S sensing performance can be understood by considering both heterojunction and chemical interaction effects. The CuO–SnO₂ interface forms a p–n heterojunction, which modulates the depletion layer and enhances resistance variation upon gas exposure. More importantly, CuO can react with H₂S to form copper sulfide species, which possess higher electrical conductivity. This reaction leads to a pronounced change in the local electronic structure and contributes to large resistance modulation. After removal of H₂S, reoxidation of

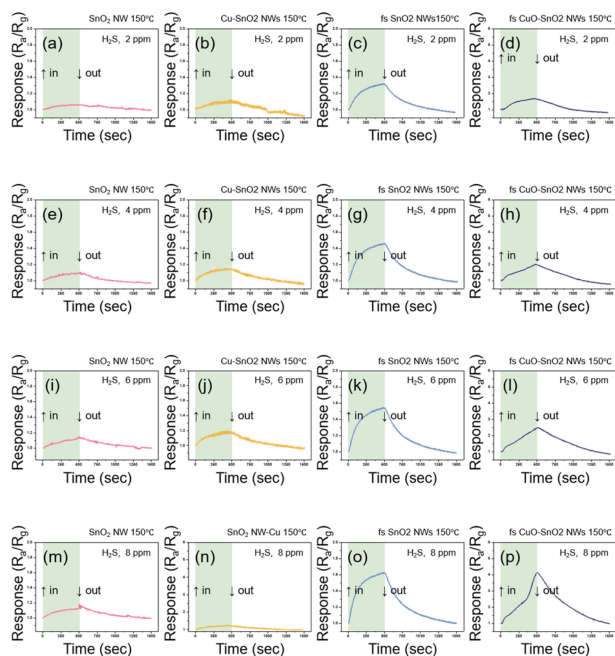


Fig. 5. Dynamic responses of pristine SnO₂ NWs, Cu-decorated SnO₂ NWs, fs-laser treated SnO₂ NWs, and fs-laser treated CuO-decorated SnO₂ NWs sensors toward different H₂S concentrations at 150°C. (a–d) 2 ppm, (e–h) 4 ppm, (i–l) 6 ppm, and (m–p) 8 ppm H₂S.

copper sulfide in air allows the sensor to recover its baseline resistance [22–24].

In addition, fs-laser irradiation likely increases surface defect density and modifies oxygen-related species, as suggested by XPS analysis. These defect sites can promote adsorption and charge transfer processes, further amplifying the sensing response. Therefore, the superior performance of the fs-laser treated CuO-decorated SnO₂ NWs sensor can be attributed to the combined effects of heterojunction modulation, chemical interaction with H₂S, and defect-assisted surface reactions [25,26].

4. CONCLUSIONS

In this study, fs-laser irradiation was employed as a surface engineering process to modify CuO-decorated SnO₂ NWs for H₂S gas sensing. The fs-laser treatment preserved the nanowire morphology while altering the surface chemical states and defect-related oxygen species. CuO species were successfully introduced onto the SnO₂ NWs surface, forming CuO–SnO₂ heterojunctions.

Gas sensing measurements demonstrated that the fs-laser treated CuO-decorated SnO₂ NWs exhibited improved responses toward H₂S compared to pristine and CuO-decorated SnO₂ NW. The optimal performance was observed at 150°C,

where a response of 5.38 was achieved toward 10 ppm H₂S. At this temperature, the response increased monotonically with the H₂S concentration, from 1.68 at 2 ppm to 2.02, 2.47, and 4.12 at 4, 6, and 8 ppm, respectively. The corresponding response and recovery time toward 10 ppm H₂S was 462 s and 850 s, respectively. The enhanced sensing behavior is attributed to the combined effects of p–n heterojunction modulation and the reversible chemical interaction between CuO and H₂S, along with defect-assisted surface reactions induced by fs-laser processing.

These results indicate that femtosecond laser irradiation can serve as an effective post-treatment strategy to tune the surface states of metal oxide nanostructures and improve gas sensing performance without altering the bulk structure. This process-oriented approach provides a feasible route for performance optimization of chemiresistive gas sensors.

CRedit Authorship Contribution Statement

Sangwoo Kim: Resources, Methodology, Conceptualization.

Jiyeon Shin: Writing – original draft, Resources,

Methodology, Conceptualization. **Jimyeong Park:** Data

curation, Validation. **Taehoon Park:** Data curation,

Visualization. **Jae Jin Sim:** Data curation, Visualization.

Changyu Kim: Investigation, Formal analysis. **Jae-Hyoung**

Lee: Writing – review & editing, Visualization, Validation,

Supervision. **Myung Sik Choi:** Writing – review & editing,

Supervision, Funding acquisition.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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