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Tapered Optical Fiber for Hydrogen Sensing Application Based on Molybdenum Trioxide (MoO_3) †

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Abstract: In this work, molybdenum trioxide (MoO_3) was synthesized and deposited on tapered optical fiber using the drop-casting technique for hydrogen (H_2) detection at room temperature. A transducing platform in a transmission mode was constructed using multimode optical fiber (MMF) with a 125 μm cladding and a 62.5 μm core diameter. To enhance the evanescent light field surrounding the fiber, the fibers were tapered from 125 μm in diameter to 20 μm in diameter with a 10 mm waist. The microstructures and chemical compositions of the fabricated sensor were analyzed by field emission scanning electron microscopy (FESEM), energy dispersive X-ray (EDX), differential X-ray (XRD), and atomic force microscopy (AFM). In addition, the gas detection properties of the fabricated sensor were studied by exposing it to various concentrations of hydrogen gas from 0.125% to 2.00%. As a result, the sensitivity, response, and recovery time were 11.96 vol%, 220 s, and 200 s, respectively. Overall, the fabricated sensor exhibits good sensitivity as well as repeatability and stability for hydrogen gas detection.

Keywords: hydrogen (H_2) gas; tapered optical fiber; evanescent wave; molybdenum trioxide (MoO_3) and drop-casting technique

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1. Introduction

Hydrogen gas has a wide explosion range (4–75%), small ignition energy (0.02 mJ), and a high flame propagation speed [1]. It can leak out of storage as H_2 because its molecule size is too small [2]. These reasons underscore the importance of H_2 detection to help reduce the risk of human exposure to the gas [3]. The fact that the optical signal is immune to electromagnetic interference, non-inductive with less attenuation than the electrical signal, is ideal for gas detection applications [4]. Therefore, the optical sensors rely on optical fibers, which have unique characteristics, such as lightness, small size, electromagnetic interference resistance, instability, and stiffness in harsh environments [5]. Due to their peculiar properties, optical fibers are perfect candidates for detection in harsh environments. In parallel, depositing the nanostructured metal oxide detection layer can provide new optical detection properties for gas [6]. Optimizing the analytical surface interaction contributed by the higher surface-to-volume ratio of nanostructures improves the performance of optical gas detection. So, the main motivation to improve H_2 sensing is due to

the health and safety concerns that have arisen with the vast popular use of H_2 and its associated technologies.

In recent years, improved nanotechnology promises an astonishing evolution in gas sensor design and capabilities. Various hydrogen oxide sensors, including SnO_2 [7,8], ZnO [9,10], and Wo_3 [11,12], as well as their mixtures, were examined. However, the response speed and sensitivity are still not satisfactory. Recent work aims to explore another metal oxide that can provide better cross selectivity and response. Molybdenum trioxide (MoO_3) shows promise in energy storage, catalysis, and gas detection applications [13]. Recently, the experiment showed that the interaction of optical fiber-based MoO_3 with hydrogen is highly sensitive, fast response to ant time, and an efficient eye-readable gas detection technology has been developed as a bio complement of the conventional H_2 sensors [14].

In this work, tapered optical fiber coated with MoO_3 is used to detect hydrogen gas. It also explains the possible interaction mechanisms between the MoO_3 sensing layer and hydrogen gas.).

2. Experimental

2.1. Tapering Process of Optical Fiber [15]

The H_2 gas sensor was fabricated utilizing a multimode fiber (MMF) with cladding and core diameters of 125 μm and 62.5 μm, respectively, as a conversion platform. A Vytran glass (Vytran GPX-3400) processing machine was used for the taper. To obtain the desired geometry of the tapered fibers, the machine heating and pulling the fiber uses graphite filament as a heater. The MMF was tapered from the cladding with a 125 μm to a waist diameter of 20 μm, a waist length of 10 mm, and a top and bottom taper of 5 mm. Figure 1 depicts the fabricated tapered optical fiber with the tapered region. According to [15], this tapered shape responds significantly between the gas sensing layer and the evanescent field.

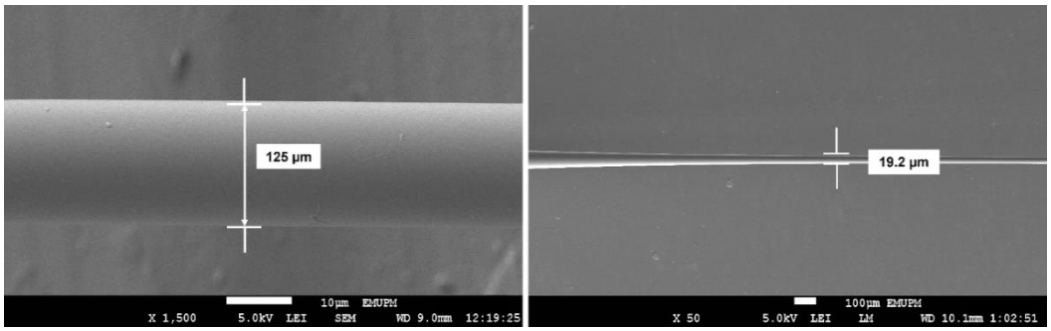

Figure 1. FESEM image of the multimode optical fiber (MMF) before and after tapering.

2.2. Fabrication of MoO_3 on Tapered Optical Fiber

Molybdenum trioxide (MoO_3) powder was synthesized by the simple solid decomposition method [16]. A 2.50 g of ammonium heptamolybdate tetrahydrate was taken and ground in suspension for one hour and annealed in an alumina crucible at 500 °C for three hours in the air. The resulting product was washed with distilled water and dried in an air oven. Prepared MoO_3 (0.25 g) was dispersed in 10 mL of deionized water to get a milk-white suspension after ultrasonic treatment for 30 min.

The tapered optical fiber was coated with MoO_3 was done using the drop-casting technique. Using a micropipette, drooped about 15 μL of the mixture to the tapered base and heated the sample in the oven at 80 °C for 30 min to complete evaporation of the aqueous medium.

The experimental equipment's of the optical gas sensing system includes: a light source with a wavelength of 360 to 2500 nm (tungsten halogen, HL-2000, Ocean Optics, Dunedin, FL, USA), spectrophotometer (USB 4000, Ocean Optics, Dunedin, FL, USA), the detection range of the optical absorbance spectrum is 200–1100 nm. Use standard (S.M.A.

fiber) cables to connect the light source to the fiber optic. The inside diameter of the S.M.A. cable is 600 µm, and the FC/PC type is used as the MMF terminal. The adapter data file recognizes the standard input loss format for MMF terminated with FC/PC-SMA cables determined to be 1.4 dB. Figure 2 shows the experimental installation of sensor H_2. Before the computer-controlled mass flow controller performs the gas purification at a gas flow rate of 200 sccm, the MoO_3 coated-based sensor is placed in a closed gas unit. The H_2 gas is purified by pure synthetic air to achieve the concentration range from 0.125% to 2.00%. The dynamic response and cumulative absorbance were determined while purging synthetic air.

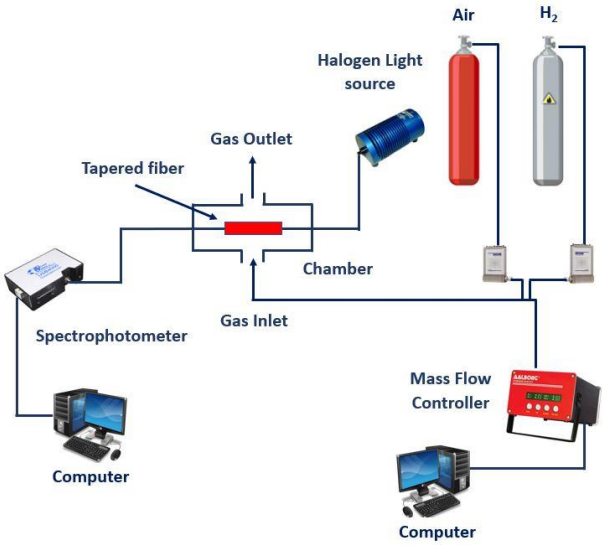

Figure 2. H_2 sensor experimental setup.

2.3. Structural Characterization of MoO_3

Various characterization techniques have been applied to characterize MoO_3 thin film. The morphology was observed by FESEM (Nova Nanosem 230), while the initial composition was determined by EDX analysis. The identification of the material, crystallization, and the phase transition of the MoO_3, was determined by analysis of XRD (PW 3040-Philips) and AFM (Dimension Edge with ScanAsyst).

The FESEM images demonstrate the successful deposition of MoO_3 on the surfaces of the tapered optical fibers, as shown in Figure 3a. The standard features of the MoO_3 nanoparticles are shown in Figure 3b,c. The particles agglomerated in nature after attempting to form thin plate or flake-like formations. The gas analyte will interact more effectively with this plate-like structure [17].

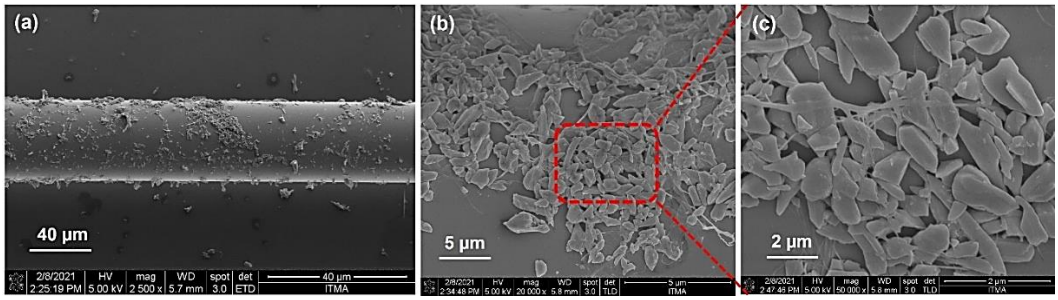

Figure 3. FESEM micrographs of (**a**) tapered MMF covered with MoO_3, (**b**,**c**) pure MoO_3.

As illustrated in Figure 4a, EDX was used to identify the elements in the synthesized MoO_3. The MoO_3 film shows the presence of Mo, O, and Si. The silicon peak (Si) was used

by the silica fibers used as a substrate. The X-ray diffraction (XRD) patterns of MoO_3 were recorded within the within 2θ range of 5 ° to 70 °, as shown in Figure 4b. The sample shows highly crystalline hexagonal and orthogonal structures, respectively, because the intensity is strong and sharp enough with narrow full-width maximum half (FWHM) of diffraction peaks such as 11.25° (100), 34.80° (101), 17.40° (110), 20.10° (200), 27.32° (210), 30.02° (300), 36.15° (310), 44.20° (320), 46.60° (410), 51.15° (002), 56.55° (211) and 58.24° (042). All the XRD peaks can be distinguished for the sample, and single-phase can be assigned to crystal structures. The prominent diffraction peak corresponding to planes (100) and (210) placed at the highest density of MoO_3.

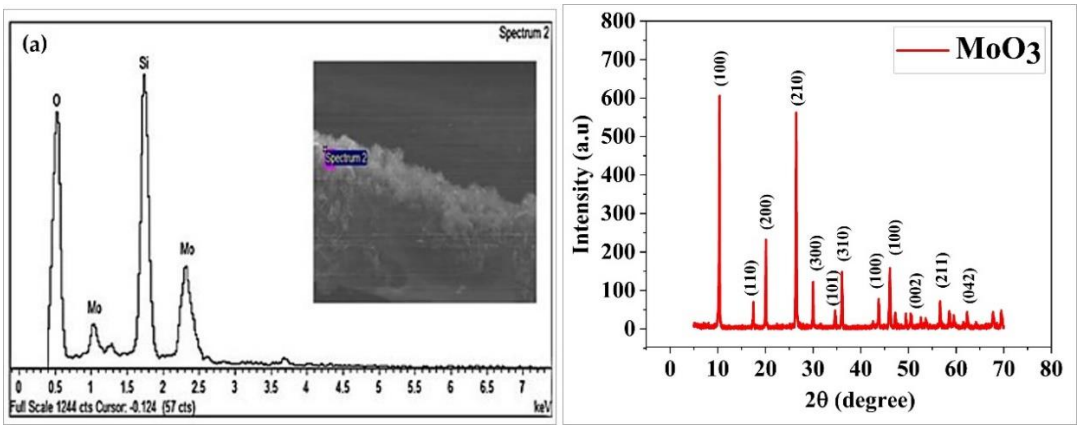

Figure 4. (**a**) EDX measurement of MoO_3 and (**b**) XRD pattern of MoO_3.

Atomic force microscopy (AFM) was conducted to characterize the average surface roughness and thicknesses of MoO_3. Figure 5a depict 3D AFM image of the MoO_3. A 10 × 10 μm section of the boundary area was scanned for the AFM analysis. The average surface roughness values were 44.98 nm. Such low roughness levels indicate that light scattering has no significant effect on sensing performance [18].

As part of this study, the thicknesses of the MoO_3 coatings were measured. Measurements were taken by covering portions of fibers with aluminium tape and then determining the differences between the thicknesses of coated and uncoated fibers. The average thickness of MoO_3 coatings was 181 nm, as shown in Figure 5b.

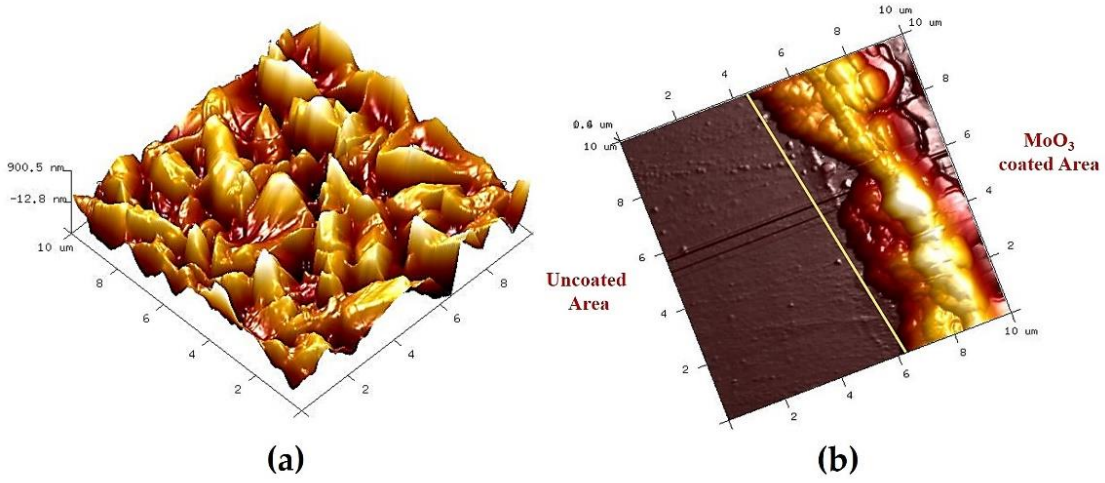

Figure 5. (**a**) 3D AFM image of MoO_3, and (**b**) 3D AFM topography images of the boundary region between the uncoated and coated fibers of MoO_3.

3. Results and Discussion

At room temperature, the absorption spectra of the developed sensor coated with MoO3 to synthetic air and 2.00% H_2. The MoO_3 coated sensor exhibits noticeable changes in absorption, as illustrated in Figure 6a, especially in the wavelength range of 550 to 850 nm. The cumulative absorbance of the MoO_3 coated sensor was analyzed to determine its performance. The dynamic response of a MoO_3 coated based sensor toward H_2 concentrations in synthetic from 0.125% to 2.00% at room temperature is shown in Figure 6b. The response and recovery times of the developed sensor were 220 s and 200 s, respectively. At 0.125% H_2, the absorbance changed by about 8%, while at 2.00% H_2, it changed by about 25%. The MoO_3 coated sensor had higher H_2 absorbance and recovery, as well as advanced compromise differences.Three cycles of 2.00% H_2 were used to test the repeatability of the MoO_3 coated based sensor. Overall, the MoO_3 coated sensor demonstrated a strong and stable absorbance response and excellent repeatability towards H_2.

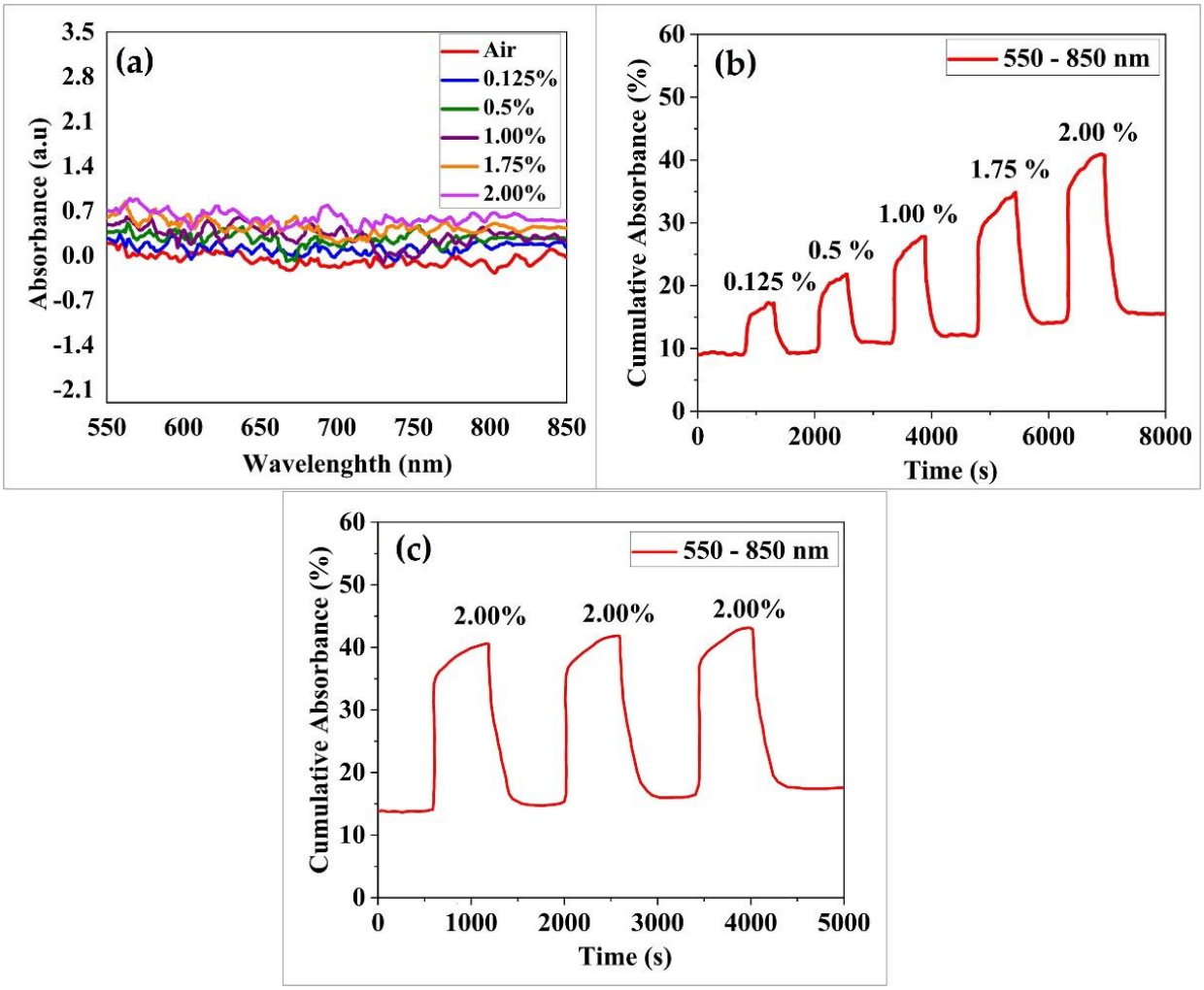

Figure 6. (**a**) The absorbance with various concentrations, (**b**) dynamic response, and (**c**) repeatability of MoO_3.

Figure 7a shows the absorption versus H_2 concentration for MoO_3 coated based sensors. The MoO_3 coated based sensors had a sensitivity of 11.96/vol% and a linearity slope of 98%. When measuring gas sensing properties, selectivity is an important key to consider. Selectivity is the most critical parameter of the sensor properties. The fabricated sensor with different gases such as ammonia (NH_3) and methane (CH_4) at a concentration of 1.00% was investigated. The MoO_3 coated based sensor had a highly NH_3 absorption

response but a substantially lower response for the other gases. Furthermore, the adsorption of MoO_3 based materials was highly selective for polar molecules such as NH_3, whereas sensitivity was low for non-polar molecules such as H_2 and CH_4 [19].

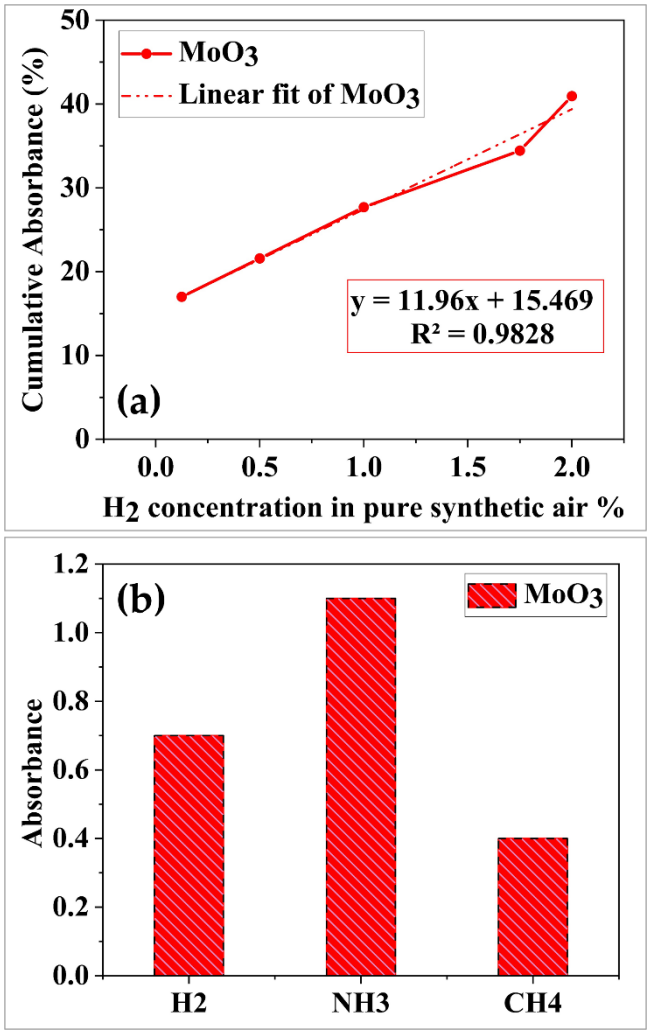

Figure 7. (**a**) Absorbance changes for MoO_3 coated based sensor at various H_2 concentrations, and (**b**) selectivity of MoO_3 coated based sensor.

4. Conclusions

In the present study, molybdenum oxide was deposited on tapered optical fiber using the drop-casting technique for hydrogen sensing decoction. The H_2 gas sensor is fabricated of the as-deposited structure, which is exposed to various concentrations 0.125–2.00% at room temperature. According to the findings, the MoO_3 coated based sensor enhanced its absorption response by 25% when exposed to 2.00% H_2 in synthetic air. The selectivity investigation indicates that the MoO_3 based optical sensor response strongly towards ammonia, methane, and hydrogen chemicals. The findings suggest that an affordable and accessible methodology may be utilized to enhance an effective, accurate, and repeatable H_2 sensor in real-world atmospheric conditions.

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