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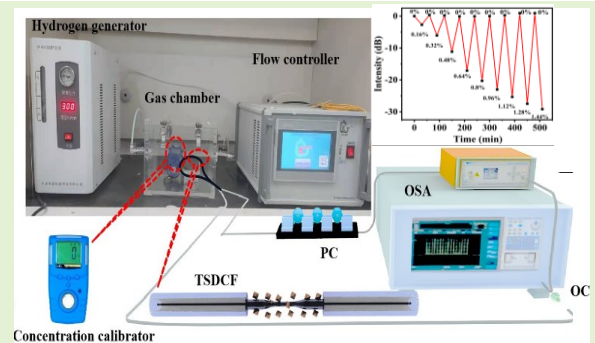
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# A sensor with coating Pt/WO<sub>3</sub> powder with an Erbium-doped fiber amplifier to detect the hydrogen concentration

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**Abstract** — A highly sensitive hydrogen sensor coated with Pt/WO<sub>3</sub> powder with an Erbium-doped fibre amplifier (EDFA) is proposed and experimentally demonstrated. The sensing head is constructed by splicing a short section of tapered small diameter coreless fiber (TSDCF diameter of 62.5 μm, and tapered to 14.5 μm) between two single-mode fibres. The Pt/WO<sub>3</sub> powder adheres to the surface of PDMS film coated on the TSDCF structure, which is sensitive to hydrogen. An EDFA is introduced into the sensor system to improve the quality factor of the output spectrum and thus improve the sensor's resolution. As the hydrogen concentration varies from 0% to 1.44%, the measured maximum light intensity variation and the sensor's sensitivity are -32.41 dB and -21.25 dB/%, respectively. The sensor demonstrates good stability with the light intensity fluctuation of < 1.26 dB over a 30-minute duration.

**Index Terms**—Hydrogen sensor, Erbium-doped fiber amplifier, Pt/WO<sub>3</sub> powder, PDMS film.



## I. Introduction

A major clean raw material and a special gas, hydrogen is widely used in petrochemical, electronic, metallurgical, food processing, float glass, fine organic synthesis industries among others. Additionally, as environmental protection has strengthened, hydrogen has become a perfect new energy source for the aerospace and automobile industries [1]. However, as early as 1990s, scientists observed the leaky nature of hydrogen, which is faster than other fuels or gases [2]. In addition, hydrogen is flammable explosive with a strong diffusibility. Therefore, the detection of low concentrations of hydrogen is essential to avoid the danger of explosion caused due to hydrogen leak during its production, storage, transportation, and usage. Hydrogen sensors (HSs) must meet few requirements including high sensitivity, rapid

response/recovery, very good selectivity, and low detection limits. In the last decades, several types of HSs have been developed including surface acoustic [3], electrochemical and thermoelectric [4, 5], and optical [6]. Among these optical fiber-based HSs, which offer unique advantages including excellent safety, smaller size, low-cost, reusability, stable and highly accurate performance, improved sensitivity, reusable, and longer transmission range, thus making them an ideal solution in many hazardous applications [7-11]. There are several fiber optic-based HSs depending on their sensing structure, including fiber Bragg grating (FBG), fibre interference, surface plasmon resonance (SPR), etc. For example, Zhang et al proposed a hydrogen sensor based on a tilted fiber Bragg grating coated with PDMS/WO<sub>3</sub> powder with a sensitivity of 0.596 dB/% for hydrogen concentrations of 0 to 1.53 % [12]. Note that, the FBG-based hydrogen sensor has the

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advantage of distributed measurement, but of lower sensitivity [13-15]. SPR fiber-based HSs have offer higher sensitivity and fast detection but at higher cost and complex production process, which limits their practical applications [16-18]. By contrast, interference-based HSs have gained more research attention because they are simple to fabricate and perform better. Interferometric fiber sensors can be divided into three categories of Mach-Zehnder interferometric (MZI) [19-25], Fabry-Perot (F-P) interferometer [26], and Sagnac interferometer [27, 28].

The principle of optical fibre hydrogen sensor is based on coating a thin layer of hydrogen sensitive material onto the surface of the fibre sensor. The hydrogen-sensitive material will change properties thus resulting in a change in sensor signal. The most commonly used hydrogen sensitive materials are Pd and  $\text{WO}_3$ . While Pd has good selectivity, its hydrogen embrittlement prevents its adhesion to optical fibres [1]. Whereas  $\text{WO}_3$  adhered well but lacks selectivity and sensitivity to hydrogen [29]. Therefore, to improve the gas sensitivity of  $\text{WO}_3$  precious metals are often added as catalysts [30]. Many papers have reported on the effect of Pt loading on the sensing properties of  $\text{WO}_3$ . For example, in 2013 Dai et al proposed an optical fibre hydrogen sensor by depositing Pt/ $\text{WO}_3$  onto FBG,

which achieved a wavelength shift  $\Delta\lambda$  of up to 536 pm at hydrogen concentration of 10,000 ppm [31]. In 2018, Li et al proposed a dual C-cavity optical fibre hydrogen sensor based on Pt/ $\text{WO}_3$  with the hydrogen sensitivity of -15.14 nm/% over the hydrogen concentration range of 0-1 % [32].

In this paper, Pt/ $\text{WO}_3$  is used as hydrogen sensitive material. The sensing head is constructed by splicing a short section of tapered small diameter coreless fibre (TSDCF) between two single-mode fibres (SMFs). A hydrogen sensitive layer is formed on the TSDCF interference structure when Pt/ $\text{WO}_3$  powder adheres to the surface of PDMS film coated on the TSDCF structure. The hydrogen sensitive layer undergoes an oxidation-reduction reaction and release heat when exposed to hydrogen, thus changing the effective refractive index of the surface of the TSDCF structure, which results in  $\Delta\lambda$  or the variation of light intensity. **The proposed sensor system is based on intensity modulation, which can have potential low cost solution by simply using photodetector to demodulate the signal. In addition, an erbium-doped fibre amplifier (EDFA) is integrated into the sensing system, which not only provides a narrow full width at half maximum, but also amplify the signal variations and thus has higher sensitivity.**

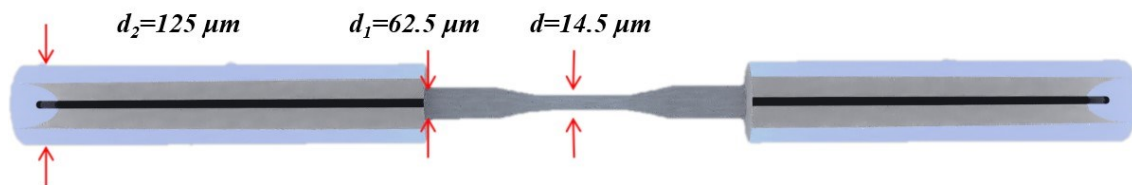


Figure 1. The TSDCF structure diagram.

## II. EXPERIMENTAL DETAILS

### A. Materials

**Synthesis of Pt/ $\text{WO}_3$ :** Pt/ $\text{WO}_3$  powder (synthesized by Hangzhou Dianzi University, China) is used as a hydrogen sensitive material to react with hydrogen, which has Pt:W molar ratio of 1:5. The polymer precursor (Sylgard 184A) and curing agent (Sylgard 184B) are also used.

**PDMS:** To obtain PDMS film, the polymer precursor and curing agent were mixed in a 5:1 ratio and stirred for 5 minutes using a magnetic stirrer at 2000 rpm.

### B. Production of sensing head

**TSDCF fabrication:** The sensor head is made by coating the surface of TSDCF with hydrogen sensitive material. Fig. 1 shows a structure diagram of the TSDCF. First, a commercial fusion splicer (Fujikura 80C) is used to fusion splice a short section of small diameter coreless fibre (CL-1010-C) between two SMFs (G652D). Second, the small diameter coreless fiber is tapered by a commercial optical coupler manufacturing system (OC-2010, JILONG) with a taper waist diameter varying from 62.5 to 14.5  $\mu\text{m}$ .

**Pt/ $\text{WO}_3$  coating:** Fig. 2 shows a schematic diagram of the procedure of coating hydrogen sensitive material Pt/ $\text{WO}_3$ . This includes two steps: 1) pour the non-solidified PDMS into the mold with the cleaned TSDCF in place to form a layer of PDMS

film on the surface of TSDCF. The thickness of PDMS film is about 1  $\mu\text{m}$ . 2) PDMS, a transparent flexible material, can firmly adhere Pt/ $\text{WO}_3$  to the surface of optical fiber. Finally, Pt/ $\text{WO}_3$  powder is adhered to TSDCF coated with PDMS film using a translation stage and then cured at 60°C for 4 h.

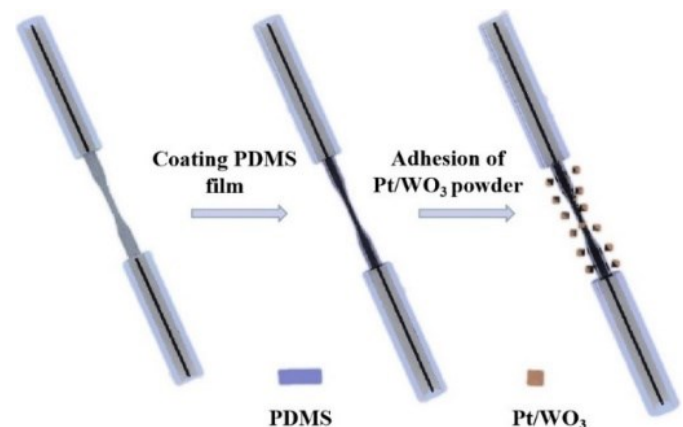


Figure 2. TSDCF structure surface hydrogen sensitive layer production process.

Figs. 3(a) and (b) is the SEM image of TSDCF structure and the SEM image of the sensor head coated with Pt/WO<sub>3</sub>, respectively. It is observed that the Pt/WO<sub>3</sub> composite film on the fiber surface is porous and rough, which is suitable for hydrogen absorption and desorption. Figs. 3(c) and (d) show the elements and specific proportions contained in the hydrogen sensitive layer. The oxidation-reduction reaction between

hydrogen sensitive layer of Pt/WO<sub>3</sub> and hydrogen can be described by the following equations [33,34]:

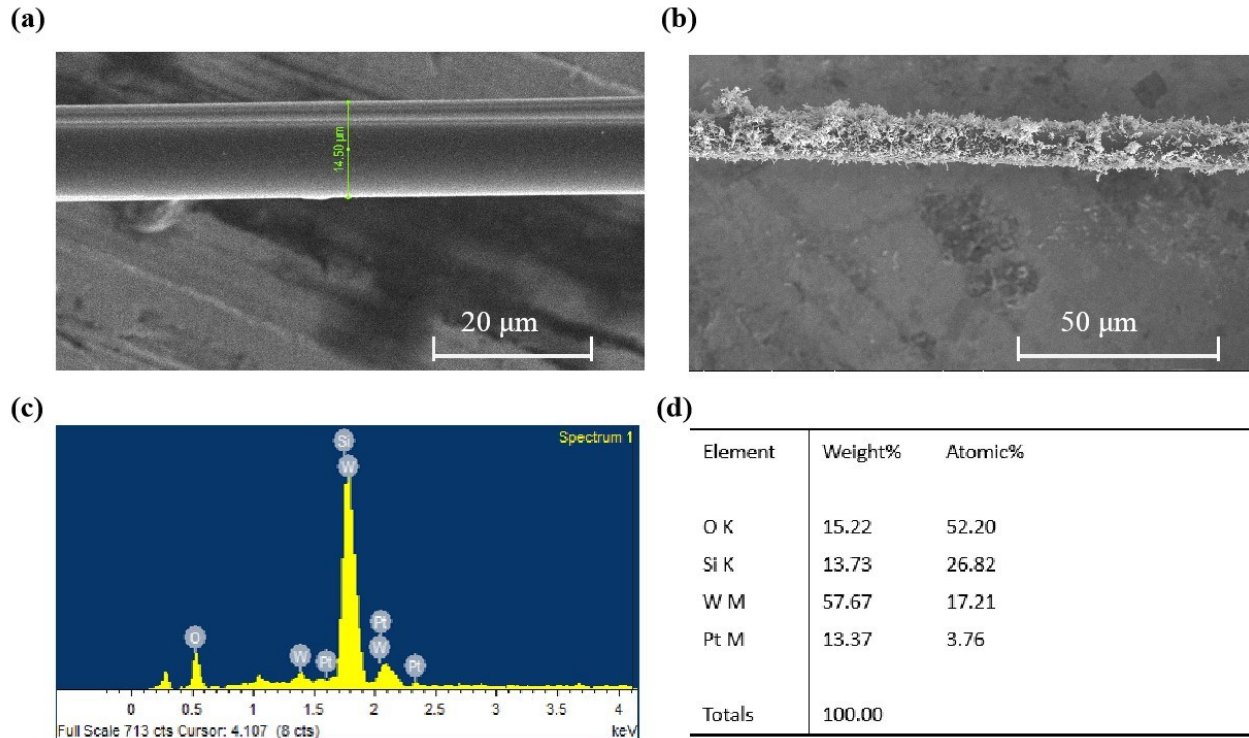
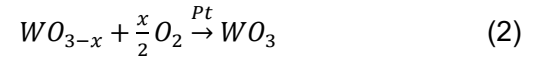
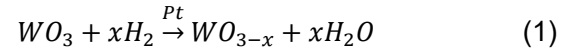


Figure 3. (a)SEM image of TSDCF structure, (b)SEM image of sensing head, (c) element analysis diagram of hydrogen sensitive layer on the surface of sensing head, and (d) the specific proportions of the elements.

### III. PRINCIPLES AND SYSTEM

When light is injected via the SMF into the TSDCF, multiple modes interference will be stimulated, which will interact with the surrounding medium via the evanescent field. A minor change in the surrounding medium will affect the output of the TSDCF, resulting in variation of the light intensity and wavelength shift. For the hydrogen sensor, due to the coating of Pt/WO<sub>3</sub>, oxidation-reduction reaction will occur when it contacts with hydrogen, which will introduce the change of light intensity and wavelength shift at the output of the sensor. In order to eliminate the difficulty of multi-band transmission spectrum analysis of hydrogen sensor and improve its resolution, an EDFA (EDFA-PA-45-6) is introduced, which is connected to a polarization controller (PC) to adjust the polarization state of light, see Fig. 4. A polarization independent isolator (PI-ISO) and a sensing head, which is located in a gas chamber, are used. The output of the sensing head is connected to a 90:10 optical coupler (OC, WIC-1X2-1550-10/90-0-A40), with the 90 and 10 % output ports are

connected to an EDFA and an optical spectrum analyzer (OSA, Anritsu, MS9710C), respectively. The experimental testbed of the proposed hydrogen sensor system is shown in Fig. 4. The sensing head is placed in the gas chamber, and the hydrogen and the nitrogen are controlled by the flow controller (KT-C4Z) to ensure the correct level of hydrogen concentration injected into the gas chamber. Hydrogen generation is provided by hydrogen generator (HP-H300). Nitrogen is stored in the nitrogen tank.



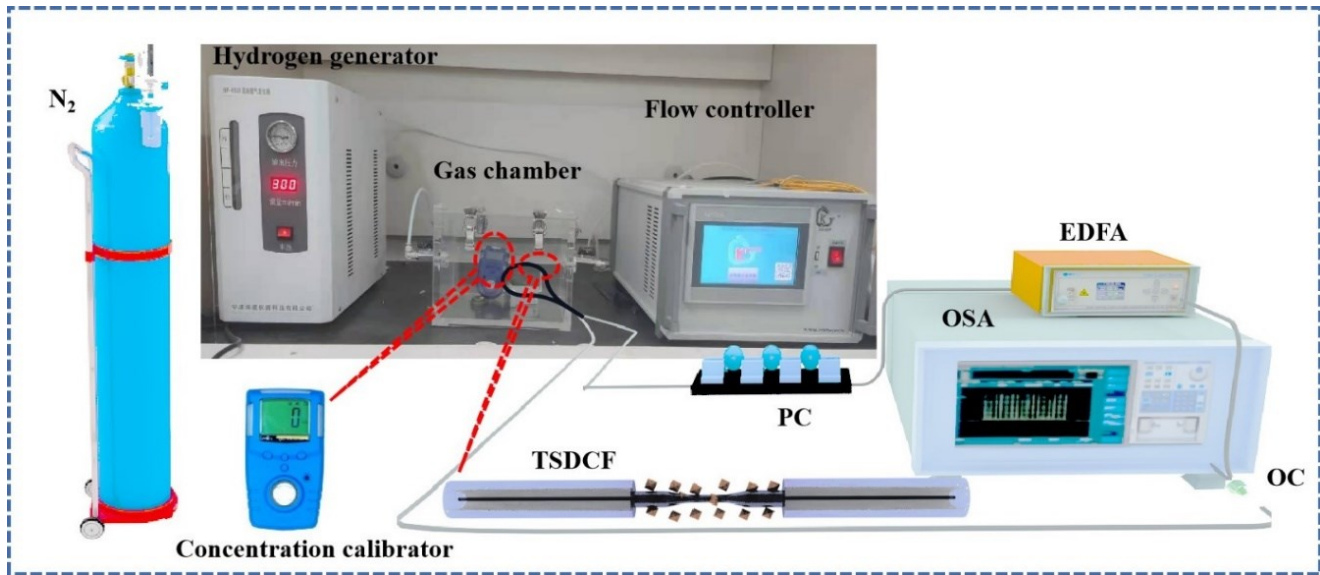


Figure 4. The experimental device of hydrogen sensor.

#### IV. EXPERIMENTAL RESULTS AND ANALYSIS

EDFA is introduced into the sensor to obtain single-wavelength transmission spectra with high quality factors (Q-factor). Fig. 5(a) shows the comparison of transmission spectra before and after the introduction of EDFA, where the red and black curves represents the transmission spectra before and after the introduction of EDFA, respectively. The optical signal-to-noise ratio (OSNR) and full width at half maximum (FWHM) of transmission spectrum change from 3.78 dB and 2.1 nm to 32.45 dB and 0.12 nm, respectively. The quality factors (Q-factor) can be calculated as follows [26]:

$$Q = \lambda / FWHM \quad (3)$$

Where  $\lambda$  is the resonant wavelength. After the introduction of EDFA, the Q-factor of the transmission spectrum of the sensor is increased by 17.5 times compared to that without EDFA.

The hydrogen concentration is measured as follow: (i) the sensing head is placed in the gas chamber; (ii) nitrogen at a flow rate of 200 ml/min is used to discharge the air within the chamber; and (iii) after all the gas in the chamber is removed by nitrogen, the hydrogen is applied to the air chamber and its flow rate is controlled at 10 ml/min. Note, a flow controller unit is used to control the concentration of both nitrogen and hydrogen within the gas chamber.

Fig. 5(b) shows the spectral response of the hydrogen sensor for different concentration rates. As can be seen from Fig. 5(b), the light intensity at the peak wavelength of 1564 nm decreases from -32.41 to -61.9 dBm when the hydrogen concentration increases from 0 to 1.44%. **When the hydrogen concentration reaches 1.44%, the OSNR of the output spectrum tends to zero. Even if the hydrogen concentration is further increased, the intensity of the output spectrum does not change significantly.**

Fig. 5(c) depicts the light intensity as a function of hydrogen concentration. Note, the rate of change of the proposed hydrogen sensor is  $\sim 21.25$  dB/%, which is significantly higher than 0.596 dB/% for the  $WO_3$  powder coated long period fiber optic fiber reported in [12]. The summary of resulted reported on hydrogen sensors together with the proposed system is outlined in Table 1. **Compared with other papers [12] and [35], using same intensity demodulation method, the proposed sensor has achieved higher sensitivity with an order of magnitude improvement. This is possibly due to: 1) the proposed TSDCF structure has very high sensitivity to surrounding environmental changes, which acts as a transducer of the hydrogen sensor; 2) the hydrogen sensitive material Pt/ $WO_3$  has a higher efficiency compared with  $WO_3$  used in the literature [12] and palladium-gold alloy in [35], because Pt has a catalytic effect; 3) the introduction of EDFA into the sensor will amplify the signal variations and thus has higher sensitivity.**

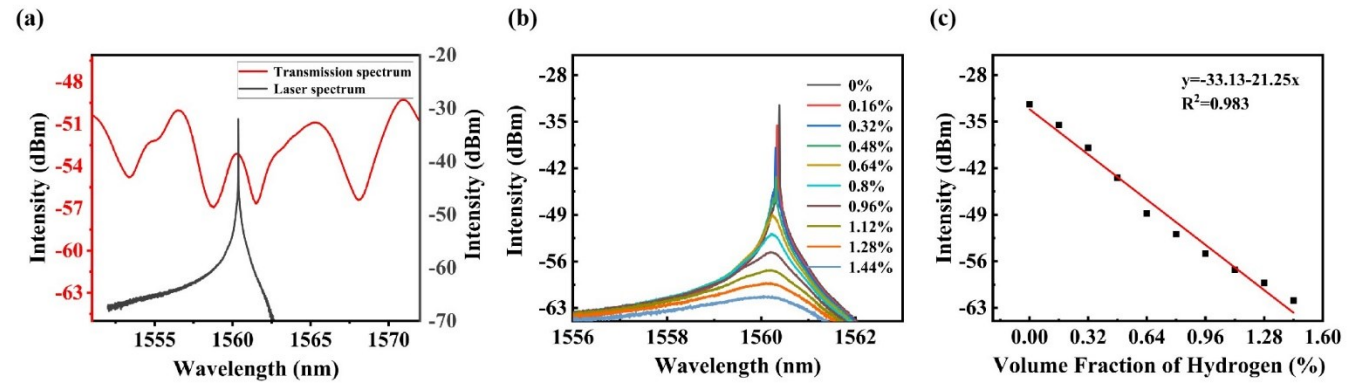


Figure 5. (a) Transmission spectrum comparison of hydrogen sensor before and after introducing EDFA, (b) Spectral response of the fibre hydrogen sensor to different concentrations of hydrogen, and (c) the relationship between the light intensity and hydrogen concentration.

TABLE 1 A SUMMARY OF HYDROGEN SENSORS FEATURES.

Method	Dynamic range (%)	Sensitivity	Reference
TFBG with PDMS/WO <sub>3</sub> composite film	0 - 1.53	0.596 dB/%	[12] (2022)
TFBG coated palladium-gold alloy	0 - 2	0.1 dB/%	[35] (2020)
SNS structure coated by Pd/WO <sub>3</sub> film	0 - 1	1.26857 nm/%	[36] (2017)
PCF coated with Pd-WO <sub>3</sub> hydrogen-sensitive film	0 - 1	1.09 nm/%	[37] (2017)
<b>Pt/WO<sub>3</sub> powder coated TSDCF sensor</b>	<b>0 - 1.44</b>	<b>-21.25 dB/%</b>	<b>This paper</b>

Next, we evaluated the stability of the hydrogen sensor by carrying out few measurements (i.e., three times) with each measurement lasting 30 minutes with an interval of 5 minutes for the hydrogen concentration of 0%. Fig. 6(a) illustrated the plots of the maximum light intensity as a function of time with

a fluctuation of 1.26 dB. The repeatability of the hydrogen sensor is shown in Fig. 6(b) with a maximum error of 1.18 dB for a set of three measurement, which is lower than the light intensity fluctuation for the hydrogen concentration is 0%.

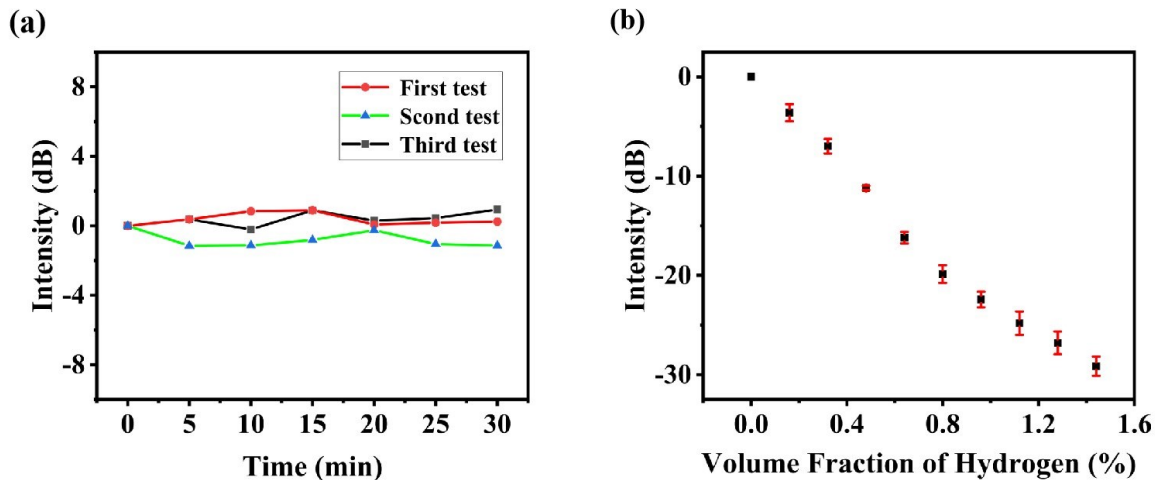


Figure 6. Hydrogen sensor: (a) the stability test for the hydrogen concentration of 0%, and (b) the repeatability plot.

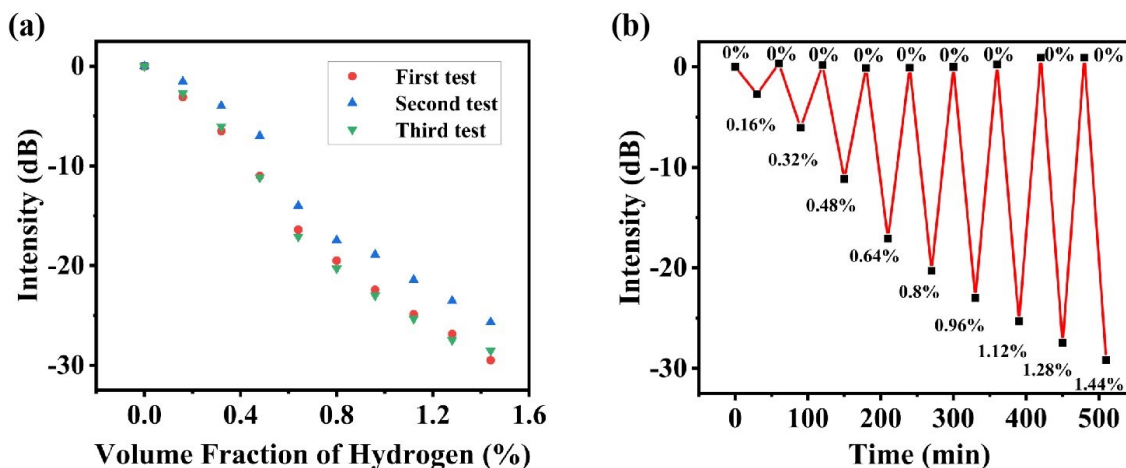


Figure 7. (a) Long term stability test of hydrogen sensor over a six-day period, and (b) light intensity changes when alternating into different concentrations of H<sub>2</sub> and fresh air in 10 cycles.

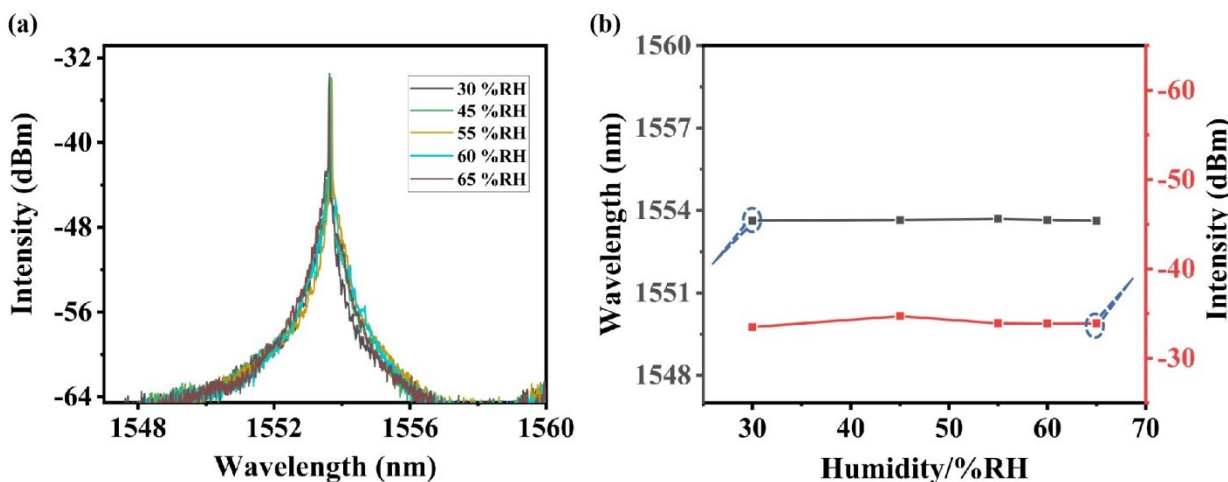


Figure 8. The results of humidity sensing at 25 °C: (a) Spectral response of the hydrogen sensor to different humidity, and (b) Variation of light intensity and wavelength with humidity.

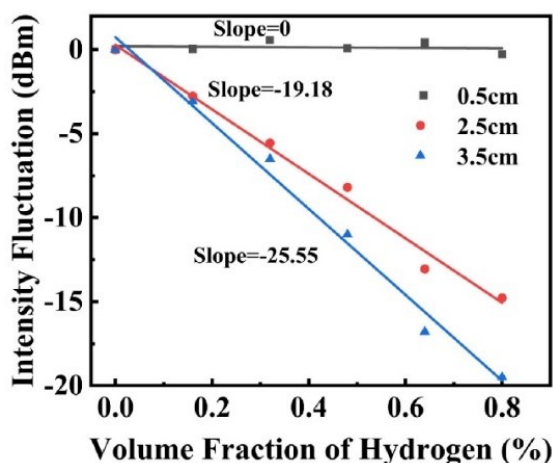


Figure 9. Comparison of sensitivity of three sensing heads with different coating lengths.

To further investigate the long-term stability of the hydrogen sensor, a series tests were carried out over six days with the results shown in Fig. 7. The proposed hydrogen sensor display

a stable intensity vs. the volume fraction of hydrogen in Fig. 7(a). According to Eqs. (2), the presence of reduction reaction will restore the hydrogen sensitive layer to Pt/WO<sub>3</sub>, which happens by exposing the hydrogen sensor to the air. Fig. 7(b) shows the measured results of 10 cycles of oxidation-reduction reaction, demonstrating a rapid recovery time of the sensor.

The influence of relative humidity (RH) on the performance of the developed hydrogen sensor has been studied as shown in Fig. 8. The experiment was carried out by varying RH from 30 % to 65 % at a fixed temperature of 25 °C. The results show that as the RH increases from 30 % to 65 %, the wavelength shift and light intensity variation are 0.064 nm and -1.23 dB, respectively. This light intensity change is less than the light intensity fluctuation of the sensor within 30 minutes under 0% hydrogen concentration. The results show that the hydrogen sensor is not affected by the change of humidity.

Finally, we investigated the influence of Pt/WO<sub>3</sub> coating length on the fiber sensor with 3.5 cm long TSDCF. Fig. 10 shows the light intensity fluctuation against hydrogen concentration for the three different coating lengths of 0.5, 2.5, and 3.5 cm. The longest coated length displays the highest sensitivity to hydrogen with the slope of -25.55 dB/%. Whereas, for the 0.5 cm long coating the intensity fluctuation of the



sensor head is independent of the hydrogen concentration.

## V. CONCLUSION

A high sensitivity TSDCF hydrogen sensor coated with Pt/WO<sub>3</sub> powder was proposed and experimentally studied. The sensor head was fabricated by attaching Pt/WO<sub>3</sub> powder to the PDMS film on the surface of TSDCF structure. We showed, a sensitivity of is -21.25 dB/% for the hydrogen concentration varying from 0 to 1.44 %. At the same time, the effects of different coating lengths on the sensitivity of the sensor were studied. Experimental results showed that the sensitivity of the sensor increased with the coating length i.e., a slope of - 25.55 dB/% for a 3.5 cm long coating.

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