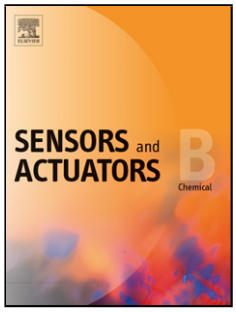

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Hydrogen Sensor Based on High-Birefringence Fiber Loop Mirror with Sol-Gel Pd/WO_3 Coating

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Highlights

- A hydrogen sensor based on high-birefringence fiber loop mirror was demonstrated, which had high-sensitivity, good repeatability, and well stability.
- The sensing head was formed by inserting a section of polarization maintaining fiber (PMF) in fiber loop mirror, which was low-cost and simple structure.
- The Pd/WO_3 coating was prepared by sol-gel method and coated on polarization maintaining fiber by dip-coating method, which was simple fabrication, low cost, and has good binding force with fiber.

Abstract

A hydrogen sensor based on high-birefringence fiber loop mirror (HBFLM) with sol-gel Pd/WO_3 coating was demonstrated. The sensing structure was formed by inserting a section of polarization maintaining fiber (PMF) with Pd/WO_3 coating in fiber loop mirror. The Pd/WO_3 coating was prepared by sol-gel method and coated on PMF by dip-coating method, which is simple fabrication, low cost, and has good binding force with fiber. When the hydrogen concentration around Pd/WO_3 coating was changed, it would induce the strain change of polarization maintaining fiber, and then shift the interference spectrum of HBFLM. Therefore, the hydrogen concentration can be measured by monitoring the wavelength shift of

the interference spectrum. Experimental results showed that the resonance wavelength had a blue shift with the increase of hydrogen concentration and the total shift of the resonance wavelength was ~2.18 nm within the concentration range of 0-1%. The sensor had simple structure, low cost, high sensitivity, good repeatability, and well stability.

Keywords: Optical fiber sensor; Hydrogen sensor; High-birefringence fiber loop mirror; Pd/WO_3.

1. Introduction

Hydrogen is a clean and reusable energy source, which has been widely used in various fields such as automobile industry, aerospace engineering, chemical processing, and so on. However, hydrogen is also extremely easy to leak out of containers and even explode in air due to its high diffusion coefficient (0.16 cm^2/s in air), low ignition energy (0.018 mJ), high combustion heat (285.8 kJ/mol), and wide explosion concentration range (4%~75%). Therefore, it is necessary to carry out warning when hydrogen concentration is one-tenth of the lower explosion limit, namely, 0.4%. Besides, it is meaningful to monitor the variation tendency of hydrogen concentration at the range of 0-1% and give an effective guidance for taking precautions.

Due to the advantages of optical fiber sensor, such as small size, immune to electromagnetic interference, intrinsic safety, and remote sensing, optical fiber hydrogen sensor has been proposed early in 1999 [1], which is based on evanescent field principle [2]. Although such sensor is the most common one in practical application, the hydrogen concentration is deduced by monitoring the transmitted intensity of light source, which is easily interrupted by external disturbances. Besides, this core-exposed method to generate evanescent field may weak the strength of sensing head, and then influence the mechanical stability of hydrogen sensor. Recently, with the further development of optical fiber technology, optical fiber hydrogen sensors have progressed rapidly and attracted great attentions. Many wavelength-demodulated optical fiber hydrogen sensors have been demonstrated, such as fiber Bragg grating (FBG), Surface plasmon resonance (SPR), and interferometer. For the FBG based hydrogen sensor [3, 4], it present the particular advantages of distributed measurement and small size, but its sensitivity is still low even some special techniques, such as side-polished, chemical etching, and tapering, are used [5-7]. For the SPR based hydrogen sensor, it receives continuously growing attentions due to its advantages of high sensitivity and rapid detection [8-10], but the production requirement and cost of the metallic film are relatively high. On the other hand, hydrogen sensors based on optical fiber

interferometers have been much more popular, which include Mach-Zechnder interferometer [11-13], Fabry-Perot interferometer [14-16], and Sagnac interferometer [17-19]. Compared to other sensors, optical fiber interferometers have high sensitivity and flexible structure. However, the interference spectrum of hydrogen sensor based on Mach-Zehnder interferometer is usually confusing due to multiple modes interfering with each other [11-13]. Hydrogen sensor based on Fabry-Perot interferometer has drawbacks in practical application, such as restriction on cavity size owing to coupling loss, large transmission loss, and offset of fiber end faces [14-16]. In contrast, hydrogen sensor based on Sagnac interferometer, also known as fiber loop mirror (FLM), has more simple structure and is easy to fabricate [17, 18]. In 2013, Y. Kim et al. demonstrated an optical fiber Sagnac interferometer hydrogen sensor with a Pd-coated PMF [17]. At a hydrogen concentration of 4%, the interference spectrum showed a wavelength shift of ~2.48nm. But the coating of pure Pd is easy to fall off, so its repeatability and long-term maintenance are limited. In 2015, Y. H. Yang et al. demonstrated another fiber Sagnac interferometer hydrogen sensor based on a polarization-maintaining photonic crystal fiber with Pd/Ag composite film [18]. The shift of interference spectrum was ~1.310 nm within the hydrogen concentration range of 0-1% and the sensor sensitivity was ~131pm/% within 1-4% hydrogen concentration. But the cost of polarization-maintaining photonic crystal fiber and the fabrication cost of Pd/Ag film are all high.

In this paper, a hydrogen sensor based on high-birefringence fiber loop mirror (HBFLM) with Pd/WO_3 coating was proposed. The Pd/WO_3 fabricated by sol-gel method acted as the sensitive material to the ambient hydrogen. Compared to pure Pd film, the binding force between the film and fiber can be increased; while compared to pure WO_3 film, the hydrogen selectivity of sensor can be enhanced. The reason is due to the difference of the reaction mechanisms: 1) the Pd in the Pd/WO_3 coating works as catalyst, which helps to dissociate hydrogen, and therefore promote the chemical reaction between WO_3 and hydrogen; 2) while in case of Pd layer, the sensing mechanism is based on phase transition of PdHx. Since α and β phase of PdHx have quite different structure, mechanical mismatch during the phase

transition cycle will generate dislocations in the coating, and finally cracks arise [19]. Besides, the influence of other interfering gases to the sensitivity of our proposed hydrogen sensor is very small and can be neglected [20, 21].

By coating Pd/WO_3 film on polarization maintaining fiber (PMF), the interference spectrum of HBFLM will be shifted with hydrogen concentration, due to the strain change of PMF. As a result, the hydrogen concentration can be measured by monitoring the wavelength shift of the interference spectrum at constant temperature of 20°C and constant humidity of 40%. In this case, the experimental results demonstrated that the sensitivity of the hydrogen sensor can reach -2.18nm/% (R^2=0.968) within the hydrogen concentration range of 0-1%.

2. Operation Principle

Fig. 1 shows the structural configuration of the HBFLM, which consists of a 3-dB optical coupler (OC) and a section PMF. Both ends of the PMF are spliced to conventional single-mode fiber (SMF). The input light is split into two beams propagating clockwise and counterclockwise by the 3dB coupler. Then the two beams are recombined at the coupler and interference appears due to the birefringence property of the inserted PMF. The transmission spectrum of the HBFLM can be described as [22]:

$$T(\lambda)=\left[\sin(\theta_1+\theta_2)\cos\left(\frac{\pi BL}{\lambda}\right)\right]^2 \tag{1}$$

where θ_1 and θ_2 are the angles between the light at both ends of the PMF and the fast or slow axis of the PMF, respectively, and B, L and λ are the birefringence, the length of PMF and the wavelength, respectively. The wavelength spacing between the transmission dips can be written as [22, 23]:

$$\Delta\lambda \approx \frac{\lambda^2}{BL} \tag{2}$$

Fig. 2 shows the interference spectra of the HBFLM for different B and L of the PMF. It can be observed that when other parameters are fixed, the birefringence B of the PMF decreases as the period of the transmission spectrum increases, and the length L of the PMF is inversely proportional to the period of the transmission spectrum. When a strain is applied to

the HBFLM, the birefringence and the fiber length of the PMF will change, and the resonance wavelengths will shift.

The metal Pd has high hydrogen absorption capacity and high selectivity to hydrogen, while the WO_3 film has better hydrogen sensitivity, good adhesion and mechanical properties. When Pd/WO_3 contacts with hydrogen, the absorption and decomposition of hydrogen takes place under the catalysis of Pd. Hydrogen atoms are transferred to the surface of WO_3 by Pd and then spread along the surface of the inner holes of WO_3, which cause the expansion of the Pd/WO_3 coating and imposing strain on the PMF coated Pd/WO_3 and associate with the change of hydrogen concentration. Therefore, hydrogen concentration can be measured by monitoring the wavelength shift of interference spectrum of the HBFLM, and the interference spectrum has a blue shift.

3. Experimental Setup

Fig. 3 shows the experimental configuration of the proposed hydrogen sensor, which consists of an amplified spontaneous emission source (ASE), an optical spectrum (OSA) with spectral resolution of 0.02nm, and a HBFLM. The HBFLM consists of a 3-dB optical coupler (OC) with low insertion loss, a PMF with length of 15 cm and Pd/WO_3 coating. A tank of pure nitrogen and a tank of nitrogen and hydrogen mixture with standard 3.5% hydrogen concentration were prepared for hydrogen sensing experiments. First, the vacuum pump was used to remove the residual gas in the pipeline. Then, opening the valves. Here, the gases from the tanks were controlled by the gas-flow meters and then mixed into the measurement chamber. Finally, the combination of gases formed a stable atmosphere of hydrogen with certain concentration and the sensing structure perceived the change of the hydrogen concentration in the chamber.

The hydrogen sensitive material in our sensor is Pd/WO_3, which can be prepared by various methods [24, 25]. In this paper, the sol-gel method is chosen [26] due to its low cost and simple fabrication. The specific preparation process is shown in Fig. 4: (1) Chemical reactions

between tungsten powder and hydrogen peroxide; (2) Removing of redundant tungsten powder and other impurities by filtering process, through which we can obtain the polymers tungstate solution, as shown in Fig, 5(a); (3) Removing of some insoluble impurities ; (4) Adding alcoholic solution to the above solution, along with the evaporation process, to obtain WO_3 sol, as shown in Fig. 5(b); (5) Doping $PdCl_2$ to WO_3 sol to obtain the Pd/WO_3 sol, as shown in Fig. 5(c); (6) Coating the Pd/WO_3 sol to the PMF surface, by using the dip coating machine; (7) Improving the performance of the film and enhancing and binding force between the film and fiber by conducting a thermal treatment under temperature of 100°C for 1 h. After the above steps, a brownish-yellow sol with homogeneous stabilization and a PMF coated with smooth film are obtained. During the experiment, several samples with different doping ratios were prepared. With the increase of the doping amount, the viscosity of the sol increased and more ethanol was needed to dissolve, which indicated that the structure of the sol was more unstable and had a tendency to rapidly solidify. The quality and stability of the samples with different doping ratios and the quality of the coating were evaluated. As a result, for doping ratio below 1:100, the coating quality is good. But at the same time, the high amount of doping was helpful to improve the response time and selectivity of hydrogen sensor. Therefore, we chose 1:100 as the optimum molar ratio of Pd:W in this manuscript by synthetically considering the coating quality and sensor properties.

During experiments, the chamber was purged with pure nitrogen (0% hydrogen concentration) before filled with hydrogen of different concentrations. Then gas with standard hydrogen concentrations of 0.2%, 0.4%, 0.6%, 0.8% and 1% was flowed into the chamber. The resonance wavelength of the interference spectrum was recorded when the spectrum became stable. It usually takes ~1 min. The main reason is that in our experiment the hydrogen concentration is controlled by mixing nitrogen and certain hydrogen with constant concentration of 3.5% as mentioned above, and the mixed hydrogen with specific concentration is gradually transmitted to sensor probe and the hydrogen concentration around the sensor probe is gradually varied. In practical test, this time delay can be largely decreased.

4. Results and Discussions

Fig. 6(a) exhibits interference spectra of the HBFLM with Pd/WO_3 coating under different hydrogen concentrations. It is observed that the resonance wavelength has a blue shift with the increase of hydrogen concentration and the total shift of the resonance wavelength is ~2.18 nm. Fig. 6(b) gives the tendency of the resonance wavelength versus hydrogen concentration. The good polynomial fit shows that the sensor has a high hydrogen sensitivity, each resonance wavelength shifts averaging ~ 2.18 nm/% in the concentration range from 0-1%. The error bars for the six data points in Fig. 6(b) have been showed in Fig. 6(c). The output result (wavelength) of hydrogen sensor for each hydrogen concentration is change in a certain range, which is mainly due to the interferences of surrounding temperature, humidity, vibration, and the concentration error of measured gas. Compared to Ref. [17, 18], the proposed sensor was low cost, simple fabrication, and has a relatively high sensitivity.

It should be mentioned that the sensitivity decreases as the hydrogen concentration increases which may be due to the hydrogen absorption of Pd/WO_3 reach to saturation when the hydrogen concentration is too high or ambient temperature changes. On the other hand, it is possible that the effective refractive index of the hydrogen sensitive material decreases as the hydrogen concentration increases, and this change will cause the resonance wavelength of the HBFLM move to the short wavelength direction. This result not only improves the sensitivity of the sensor, but also causes the nonlinear of sensor. In the future, to enhance the sensitivity, stability, and linearity of hydrogen sensor, we will try another materials, such as SiO_2, WO_3, Fe_2O_3, ZnO, SnO_2, Pt/Al_2O_3, and so on, in place of WO_3 [27].

In our experiment, the repeatability of the sensor is shown in Fig. 7(a). We repeated the hydrogen sensing experiment three times. There are some slight measuring errors in each test, which mainly result from the room temperature fluctuation and the measuring errors of optical instruments. The stability of the sensor is also shown in Fig. 7(b). At 0.4% hydrogen

concentration, we recorded the shift of the resonance wavelength in 30 minutes with an interval of 5 minutes. The maximum error is 0.0021nm, which equivalents to the wavelength shift caused by 0.096% hydrogen concentration variation. Therefore, the small fluctuate can be neglected. Besides, in our experiment, the resolution of OSA is 0.02nm, so this fluctuate may be generated by the reading error. In a whole, the experimental results demonstrated that the sensor had high sensitivity, good stability and excellent repeatable.

The response time of the proposed sensor mainly depends on the sensitive material. In our previous manuscript, we have demonstrated that the response time of Pd/WO_3 based hydrogen sensor is no more than 33 min [28], so we have not repeated the experiments to measure the response time of the proposed sensor. What should be mentioned is that the response time may be improved by UV-light irradiation [29], doping graphene quantum dots [30], which will be carried out in our future work.

5. Conclusion

In conclusion, a hydrogen sensor with high sensitivity was demonstrated both in theory and experiment. When hydrogen concentration around the PMF with Pd/WO_3 coating was changed, it would induce the strain change of PMF, and then shift the interference spectrum of HBFLM. Therefore, the hydrogen concentration could be measured by monitoring the wavelength shift of interference spectrum. The resonance wavelength had a blue shift with the increase of hydrogen concentration. The total shift of the resonance wavelength was ~2.18 nm within the concentration range from 0 to 1%. The sensor had a number of valuable advantages, such as high sensitivity, excellent repeatability and stability, good safe performance, simple structure, low lost, suitable for practical application, and so on.

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Author Biographies

Ya-nan Zhang was born in Anhui, China, in June 1989. She received her M.A. degrees and Ph.D. degrees, respectively, in 2012 and 2015 from the College of Information Science and Engineering, Northeastern University, Shenyang, China. Now she is working in Northeastern University as an associate professor. Her research interests include optical fiber sensors, photonic crystal sensors, slow light technology and its sensing applications. She has authored and co-authored more than 30 scientific papers and conference presentations.

Yong Zhao received his M.A. and Ph.D. degrees, respectively, in precision instrument & automatic measurement with laser and fiber-optic techniques from the Harbin Institute of Technology, China, in 1998 and 2001. He was awarded a first prize scholarship in 2000 by the China Instrument and Control Society and the Sintered Metal Corporation (SMC) scholarship in Japan. He was a postdoctoral fellow in the Department of Electronic Engineering of Tsinghua University from 2001 to 2003, and then worked as an associate professor in the Department of Automation, Tsinghua University of China. In 2006, he was a visiting scholar of University of Illinois in Urbana and Champagne, USA. In 2008, he was awarded as the “New Century

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Hui-jie Peng was born in Liaoning, China, in March 1993. She received the B.S. degrees in the Mechanical College, Shenyang University of Technology, Shenyang, China in 2015. Now she is currently pursuing the M.S. degree in the College of Information Science and Engineering, Northeastern University, Shenyang, China. Her current research interests include optical fiber hydrogen sensors and photonic crystal sensors.

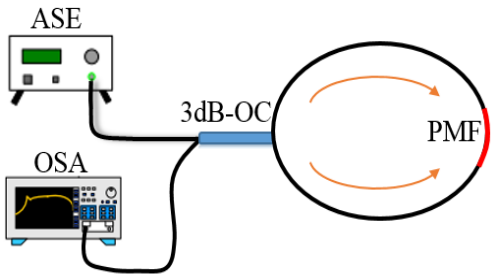

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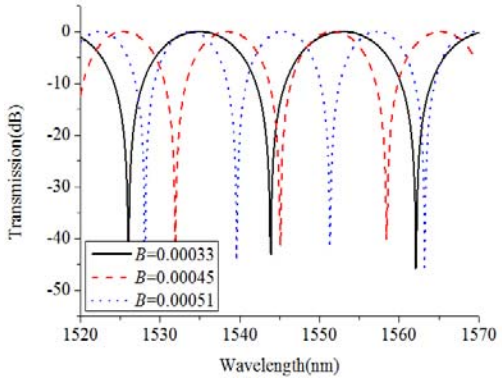
Fig. 1. Structural configuration of the HBFLM.



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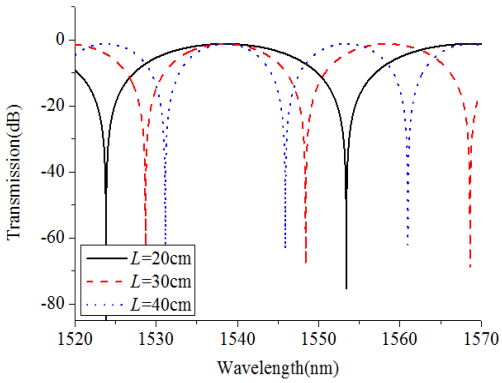

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(a) (b)

Fig. 2. Interference spectra of the HBFLM for different birefringence and lengths of the PMF.

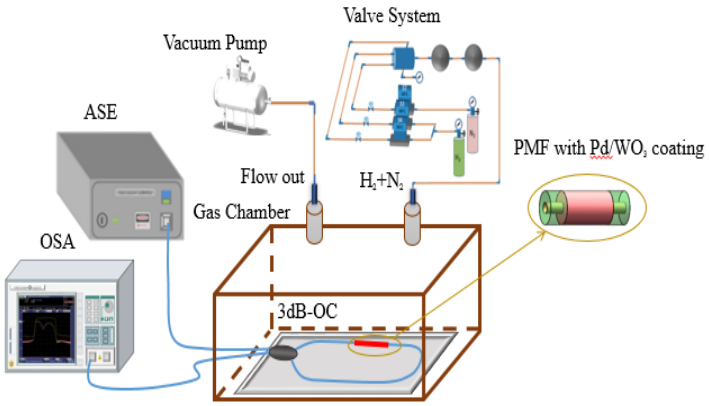

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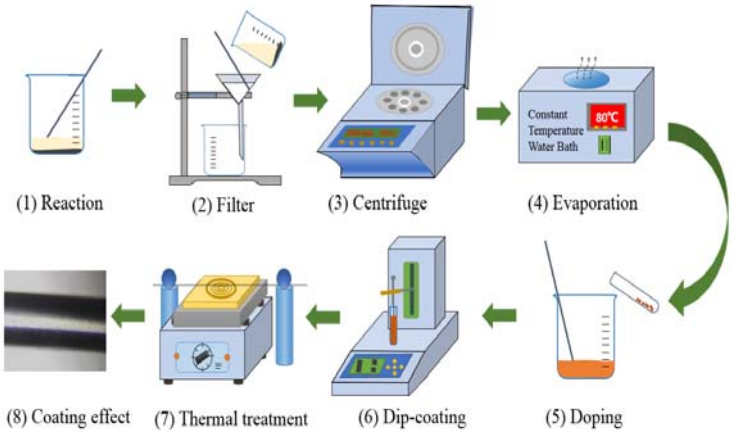
Fig. 3. Experimental configuration of the HBFLM with Pd/WO_3 coating.



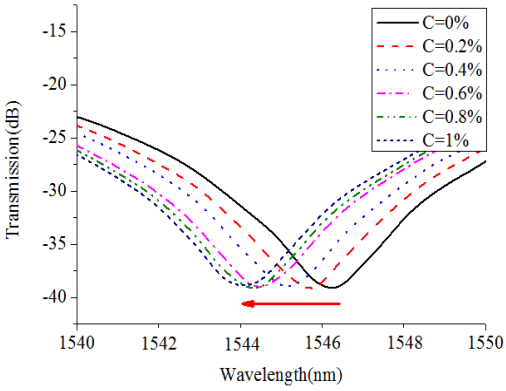
<InlineImage5>

Fig. 4. Preparation process of Pd/WO3 coating.

<InlineImage6>

(a) (b) (c)

Fig. 5. Intermediate reaction solutions in the fabrication process of Pd/WO3.



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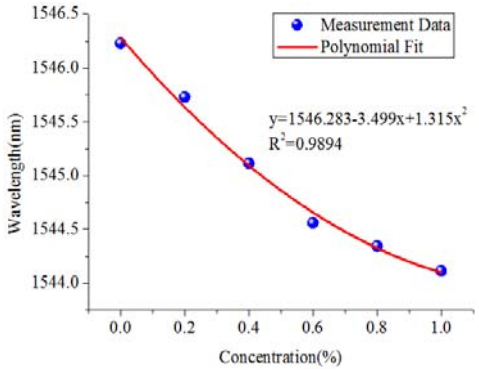

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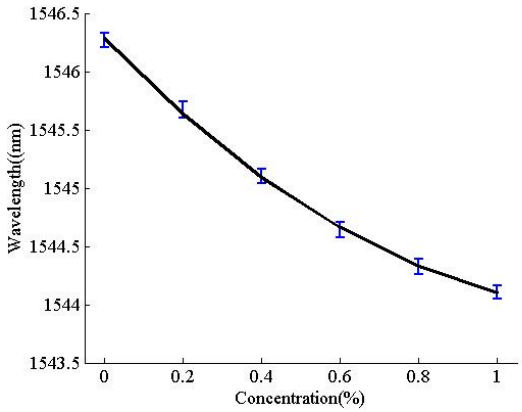
(a) (b)



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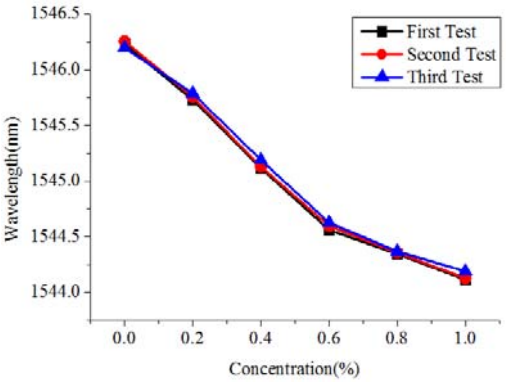
(c)

Fig. 6. (a) Interference spectrum of the HBFLM with Pd/WO3 coating under different hydrogen concentration. (b) Relation between hydrogen concentration and the resonance wavelength. (c) Error variations of six data points.



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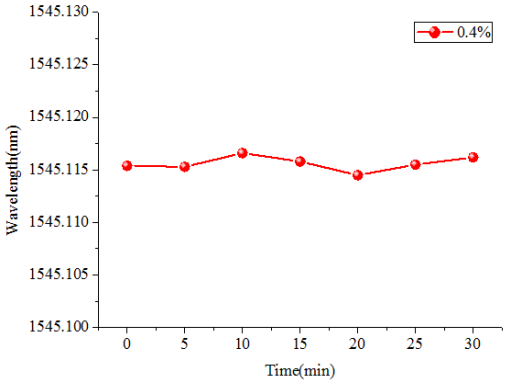

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(a) (b)

Fig. 7. (a) Repeatability of the sensor. (b) Resonance wavelength versus time. The concentration of the tested hydrogen is 0.4%.