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Enhanced sensitivity of fiber Bragg grating hydrogen sensor using flexible substrate

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ABSTRACT

A novel method was proposed to enhance sensitivity of fiber Bragg grating (FBG) hydrogen sensor in this paper. As sensing element, Pd/Ni composite film was deposited on side-face of etched fiber Bragg grating and surface of polypropylene substrate. The atomic ratio of Pd and Ni in hydrogen sensitive film was controlled at 91:9. The sensitivity of FBG hydrogen sensor is significantly improved due to the much lower Young's modulus of polypropylene substrate. FBG hydrogen sensor has 146 pm wavelength shift towards 4% hydrogen, and the sensor shows linear response and good repeatability during hydrogen concentration test. FBG hydrogen sensor has good stability even after six months, which demonstrates its great potential in hydrogen leakage monitoring.

1. Introduction

Hydrogen is a clean energy and an important chemical raw material, and it has been widely used in aerospace, fuel cells, metal smelting and chemical synthesis. However, hydrogen is dangerous because of its smallest molecule and flammable characteristic. Hydrogen leakage may take place at any facilities for hydrogen production, transportation, storage and application. Therefore, hydrogen leakage monitoring is extremely important in these facilities.

Many efforts have been carried out to explore hydrogen sensor with good performance in recent years. Optical fiber hydrogen sensor can be intrinsic safety by employing optical signal as sensing medium, and it has attracted much research interest. Several kinds of optical fiber hydrogen sensors, such as evanescent sensor [1–9], micro-mirror sensor [10,11], surface plasmon resonance (SPR) sensor [12], acoustic resonator sensor [13] and fiber Bragg grating sensor [14–24], have been proposed in recent years. Compared to other optical fiber hydrogen sensor, FBG hydrogen sensor is more suitable for distributed measurement due to its wavelength multiplexing capability. Currently, two types of FBG hydrogen sensors have been reported. The first type of FBG hydrogen sensor is based on the volume change of Pd or Pd alloys during hydrogen response. Another type is based on Pt-loaded WO_3 coatings undergoing an exothermic reaction in hydrogen atmosphere. FBG hydrogen sensor based on Pt-loaded WO_3 coating has higher sensitivity, but its performance will be decreased if there has little oxygen in ambient atmosphere. Some special facilities, such as nuclear waste tank [25], may have little oxygen in it and needs monitoring hydrogen concentration. FBG hydrogen sensor based on Pd or Pd composite film may be more suitable for hydrogen concentration measurement in such facilities. However, the sensitivity of Pd composite film coated FBG hydrogen sensor still needs to be improved to meet the demand of application. Recently, electron devices based on flexible substrate have attracted numerous research efforts due to their excellent performance [26–28]. By using flexible substrate, mechanical strength and electrical stability of the devices can be greatly enhanced. In this work, polypropylene sheet is employed as a flexible substrate to protect etched FBG because of its good stability and hydrophobicity, and the sensitivity of FBG hydrogen sensor can be increased due to its low Young's modulus. To prepare hydrogen sensor with good performance, etched FBG and polypropylene substrate sputtered with Pd/Ni composite film is proposed and investigated in this paper.

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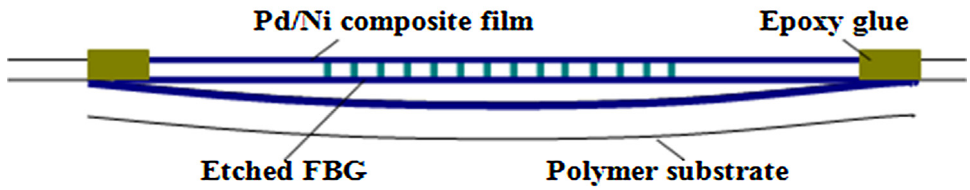

Fig. 1. Schematic illustration of sensing section of FBG hydrogen sensor.

2. Experiment

A Lambda Physik excimer laser (COMPex-150T) operating at 248 nm was used as UV light source to write FBG in single mode fiber by phase mask method [29]. FBG was annealed at 100 °C for 24 h to improve its stability. To enhance the stability and sensitivity of FBG hydrogen sensor, polypropylene sheet was employed as protective substrate, and the width and thickness of the sheet is 3 and 0.1 mm, respectively. FBG was fixed on polypropylene sheet by epoxy glue for the etching process. As reported in our previous work [30], a VHX-100 digital microscope was used to monitor the diameter of FBG during the etching process. After the etching process, the etched FBG was washed by de-ionized water and baked by infrared light for the next process. As shown in Fig. 1, two ends of polymer sheet are slightly higher than its middle section, which can ensure grating section of FBG is in suspended state. Therefore, the surface of FBG and polypropylene sheet can be completely coated with hydrogen sensitive film during the sputtering process, and the sensitivity of the FBG hydrogen sensor can be improved by this configuration.

Pd/Ni composite film was sputtered on the etched FBG and polypropylene sheet by using a BESTECH sputtering system. Firstly, 10 nm Ni film was deposited on side-face of FBG as basal layer by radio-frequency (RF) sputtering process. Secondly, 130 nm Pd/Ni composite film was sputtered on etched FBG by co-sputtering process. Thirdly, 10 nm pure Pd film was coated on Pd/Ni composite film to ensure the selectivity of hydrogen sensitive film. Under 0.5 Pa sputtering pressure of Ar, the deposition power for Pd and Ni targets are 100 and 50 W, respectively, which corresponds to deposition rate of 0.14 and 0.01 nm/s, respectively. With this sputtering process, the atomic ratio of Pd and Ni is about 91:9 in Pd/Ni composite film. During the sputtering process, the thickness of hydrogen sensitive film was monitored by quartz crystal method, which can ensure the thickness of hydrogen sensitive film. Meanwhile coatings on several polypropylene pieces were also prepared in the same run for further characterization.

The hydrogen sensing performance was carried out at room temperature of 25 °C using air as carrier gas. During hydrogen concentration characterization, the reflected wavelength is collected with a BCD-100 FBG demodulator. The BCD-100 FBG demodulator is equipped with a Fiber Fabry-Perot Tunable Filter (FFP-TF) from Micron Optics Inc., USA, which can provide high precision (1 pm) to detect a slight wavelength shift of FBG. The varying hydrogen concentrations are provided by changing flowing rate of pure H_2 injected into gas room. A commercially available RBT-6000 F/A hydrogen concentration meter (Zhuoan Company, Solid polymer electrode type) based on chemical electrics principle is connected to the gas room for calibration. The measured data is recorded by a computer connecting with the FBG demodulator for further data analysis. After hydrogen response, the morphology of hydrogen sensitive film was characterized using field emission scanning electron microscope (FE-SEM ULTRA PLUS-43-13, Zeiss, German).

3. Results and discussion

Fig. 2(a) shows the microphotography FBG after etched by HF solution. Obtained by VHX-100 digital microscope, the diameter of FBG is approximately 21 μm after the etching process. As it is

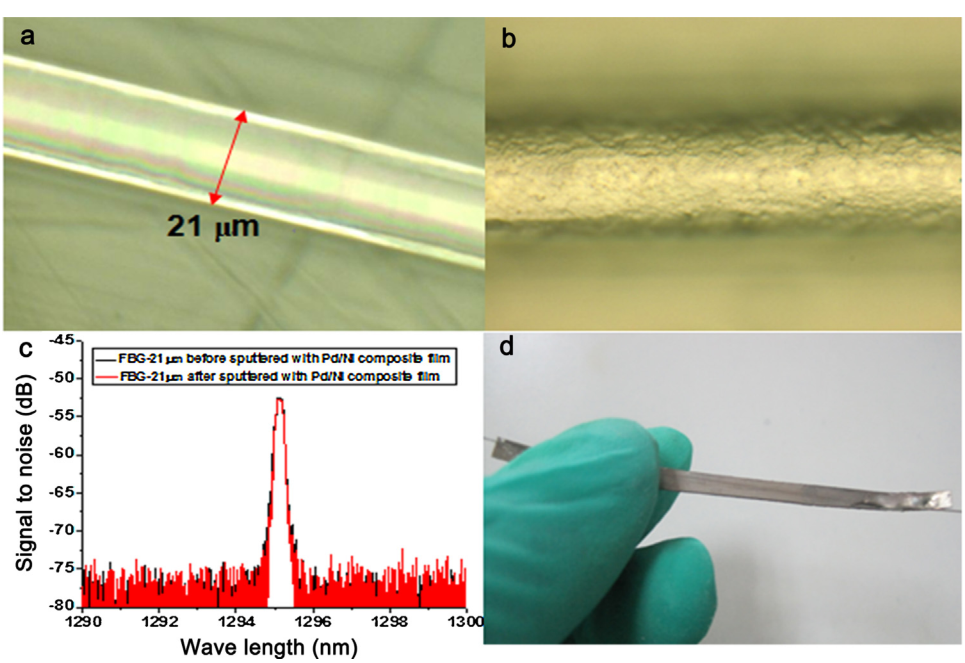

Fig. 2. Microphotography of etched FBG (a) before and (b) after sputtered with Pd/Ni composite film; (c) reflective spectrums of FBG at different stages; (d) image of sensing probe of FBG hydrogen sensor.