

Fiber in-line Fabry-Perot hydrogen sensing interferometer fabricated by femtosecond laser with Pd/Ag composite coatings

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We propose a compact hydrogen sensor, based on Fabry-Perot interferometer with Pd/Ag composite film, fabricated by femtosecond laser micromachining and thin-film coating technique. The sensing characteristic of sensor with 150-nm Pd₇₆/Ag₂₄ composite film is studied, and the device is tested to measure the hydrogen volume ratio range of 0–8%. Experimental results show that the response of the sensor is reversible, and the system shows high potential in hydrogen detection.

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Hydrogen of unusual properties including low minimum ignition energy, high heat of combustion and wide flammable range is an important industrial raw material, which is extensively used in many fields such as chemical industry, electronic industry, float glass and aerospace, etc.^[1]. Meanwhile, it is important to monitor hydrogen concentration due to its explosive nature and low ignition energy. In recent years, optical fiber sensor has attracted much attention because of its advantages, including small size, light weight, high sensitivity and immunity to electromagnetic interference for measurement of a variety of physical, chemical and biomedical parameters^[2–5]. At present, there are several kinds of optical fiber hydrogen sensors based on the palladium (Pd) thin film, such as fiber Bragg grating, in-fiber interferometer in conventional single-mode fiber, tapered fiber and photonic crystal fiber^[6–9]. Pd film is commonly used in hydrogen sensor fabrication because of the variation in its optical property with different hydrogen concentrations. However, the pure Pd film has a tendency to peel after extracting many times due to large volume expansion during PdH phase transition. In order to enhance the stability of the sensor, Pd/Ag composite film is employed in optical hydrogen sensing, which has been reported to result in greater performance^[1,10].

In previous works, a hydrogen sensor based on fiber in-line Fabry-Perot interferometer (FPI) with Pd film has been reported^[11]. To improve the sensor's repeatability, a compact hydrogen sensor based on FPI with Pd/Ag composite film is proposed in this paper. Pd/Ag composite film is deposited on the microcavity of FPI as a sensing element by magnetron sputtering process, and the sensing characteristic of FPI coated with Pd/Ag composite film has been investigated.

Figure 1 shows the structural schematic of the fiber in-line FPI deposited with Pd/Ag composite film.

The Fresnel reflection coefficients reflected from three interfaces are R_1 , R_2 , and R_3 . As the reflectivity at interface is low, the multiple reflections are neglected. The intensity of the total reflected light can be expressed as

$$I = A_0^2 \{ R_1^2 + \eta_2^2 R_2^2 + \eta_3^2 R_3^2 + 2\eta_2 R_1 R_2 \cos \{ 2k [n_{film} (d_{film} + \Delta d_{film}) + n_{cavity} ((L_1 + \Delta L_1) - 2(d_{film} + \Delta d_{film}))] \} + 2\eta_3 R_1 R_3 \cos \{ 2k [n_{film} (d_{film} + \Delta d_{film}) + n_{cavity} ((L_1 + \Delta L_1) - 2(d_{film} + \Delta d_{film})) + n_{core} (L_2 + \Delta L_2)] \} + 2\eta_2 \eta_3 R_2 R_3 \cos \{ 2k [n_{film} (d_{film} + \Delta d_{film}) + n_{core} (L_2 + \Delta L_2)] \} \}, \quad (1)$$

where A_0 is the amplitude of the incident beam, η_2 and η_3 are coupling coefficients, L_1 and L_2 are the length of the microcavity and the microcavity to the fiber end

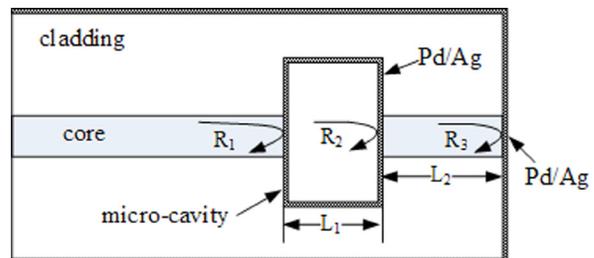


Fig. 1. Schematic structure of the fiber in-line FPI deposited with Pd/Ag composite film.

surface, respectively, d_{film} is the thickness of the Pd/Ag composite film, n_{film} , n_{core} and n_{cavity} are effective refractive indices (RI) of film, silica core and micro-cavity, respectively, and k is the wave number. ΔL_1 , ΔL_2 , and Δn_{film} are the changes in L_1 , L_2 and n_{film} due to the Pd/Ag composite film exposed to hydrogen.

Pd/Ag composite film is used as the hydrogen-sensing material due to its ability to induce strain in the FPI caused by the change in the film's volume by absorbing hydrogen. The length of FPI microcavity will be changed by the volume expansion of Pd/Ag composite film, which can cause the wavelength shift of FPI. The RI of the Pd/Ag composite film n_{film} is reduced when the hydrogen concentration is increased. The wavelength shift has correlation with hydrogen concentration due to the volume expansion^[12] and the n_{film} variation. Therefore, hydrogen concentration can be deduced by measuring the wavelength shift of FPI.

The FPI is fabricated by femtosecond (fs) laser 3D micromachining system, as schematically shown in Fig. 2, which can be divided into four parts, including the fs laser (Cyber Laser, Inc.), external optical path, CCD monitoring system and 3D moving working platform. The fs laser pulses ($\lambda = 800$ nm) of 180 fs at a repetition rate of 1 kHz are focused onto the fiber by a $10\times$ objective lens with an NA value of 0.25 and a working distance of 7 mm, and the pulse energy used is 11 μJ .

The fiber core diameter is 8.2 μm with effective index of 1.4682 (at 1550 nm). A LED is used as the light source and an optical spectrum analyzer (OSA, AQ6370B) is employed to monitor the reflection spectra of the system. The FPI microcavity with the lengths L_1 and L_2 measured to be 20 and 50 μm , respectively, by using digital microscope (VHX-100) is fabricated in the experiments, and such a device is easy to fabricate. Figure 3 shows the microscope image of the FPI fabricated by fs laser micromachining.

Ultra-high vacuum magnetron sputter system (BESTEC Germany) equipped with direct current and radio frequency sputtering sources is used to deposit the thin film. During the sputtering process, the thickness of the film is monitored by quartz crystal method. Meanwhile, thin film is deposited on an Si substrate as a standard reference sample to measure the film thickness and to improve measurement accuracy. Fig. 4 displays the scanning electron microscope (SEM) images of Si pieces coated with 500 nm Pd/Ag composite film.

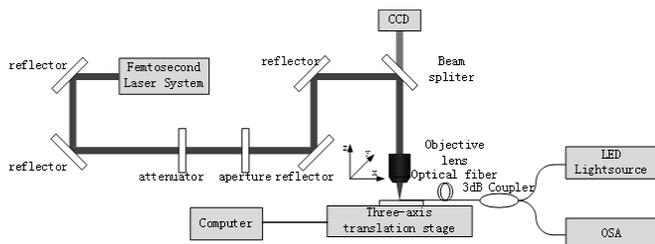


Fig. 2. System configuration of fs laser micromachining system.

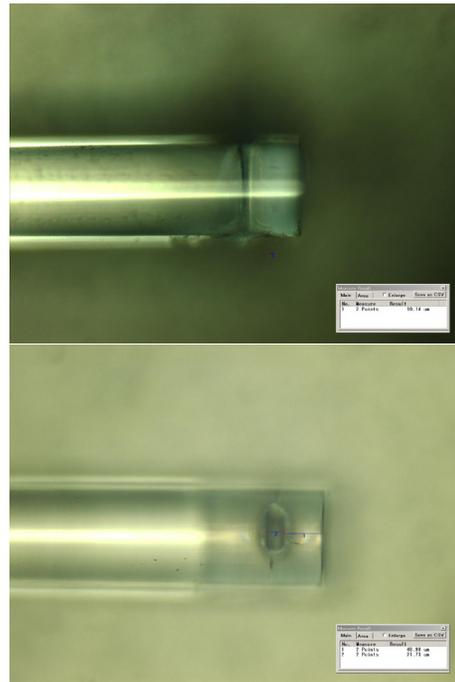


Fig. 3. Digital microscope image of FPI.

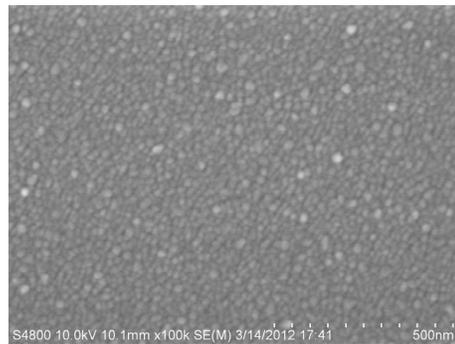


Fig. 4. SEM images of 500-nm Pd₇₆/Ag₂₄ composite film.

With this sputtering process, the atomic ratio of Pd and Ag found to be about 76:24. To ensure the selectivity of hydrogen-sensitive film, 10 nm pure Pd film is set as the protective layer outside the Pd₇₆/Ag₂₄ composite film. It can be observed that Pd/Ag composite film looks uniform and dense.

In the experiment, the thickness of the Pd₇₆/Ag₂₄ composite film is maintained at approximately 150 nm, calculated by the deposition velocity and time. The FPI sensor coated with Pd₇₆/Ag₂₄ composite film tests the hydrogen concentration response in the room temperature. The LED light source and OSA are connected to two input ports of a 3 dB fiber coupler, respectively, and the sensor in the gas chamber is connected to the output port of the coupler.

Figure 5(a) illustrates the reflection spectra of the FPI with $L_1 = 20$ μm , $L_2 = 50$ μm coated with 150 nm Pd₇₆/Ag₂₄ composite film at different hydrogen volume ratios ranging from 0 to 8%. It can be observed from Fig. 5(a) that the reflection spectrum experiences a blue shift and its intensity decreases with an increase in

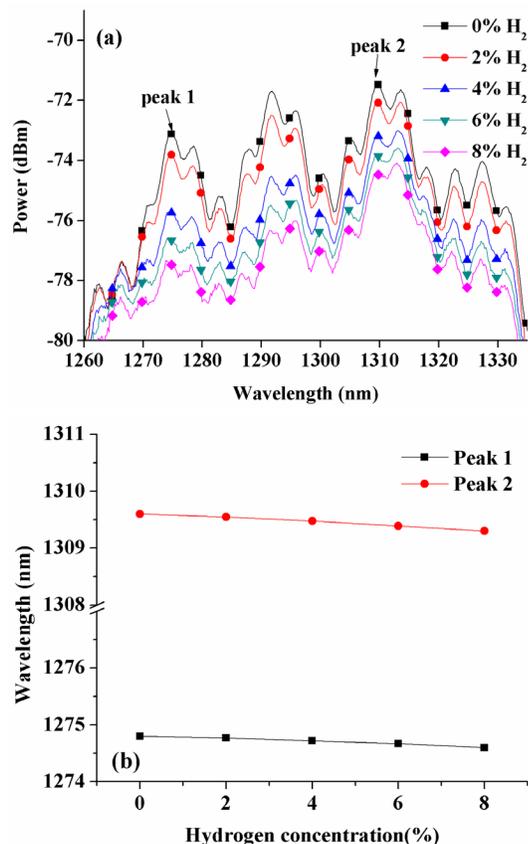


Fig. 5. (a) Reflection spectra of FPI ($L_1 = 20 \mu\text{m}$, $L_2 = 50 \mu\text{m}$) coated with 150-nm $\text{Pd}_{76}/\text{Ag}_{24}$ composite film; and (b) wavelength versus hydrogen concentration.

the hydrogen volume ratio. It has to be noted that the intensity of the peaks decreases in a nonlinear manner as the hydrogen concentration increases. Meanwhile, the reflection spectrum is changed due to change in the optical properties of the film caused by the absorption of hydrogen. 'It is mainly because the $\text{Pd}_{76}/\text{Ag}_{24}$ composite film would expand and the composite film RI can be changed with the increment in hydrogen concentration, resulting in the wavelength shift of the sensor'. The sensitivity of the peaks 1 and 2 achieved is approximately $-0.025 \text{ nm}/\%$ and approximately $-0.03 \text{ nm}/\%$ within the hydrogen concentration from 0 to 8%, respectively, as demonstrated in Fig. 5(b). After each test, air is filled in the chamber until the original spectrum is restored in several minutes. To ensure the performance of the hydrogen sensor, the measurement has been repeated several times, with which a good repeatability can be obtained. The temperature disturbance

can be decreased preferably by adopting temperature compensation method.

The performance of the sensor coated with $\text{Pd}_{76}/\text{Ag}_{24}$ composite film is comparable to the FPI with pure Pd film. Moreover, the pure Pd film is likely to fall off after testing many times, while the proposed FPI sensor with $\text{Pd}_{76}/\text{Ag}_{24}$ composite film has good mechanical performance during hydrogen response.

In conclusion, we propose and demonstrate the FPI coated with $\text{Pd}_{76}/\text{Ag}_{24}$ composite film in the microcavity. In the experiment, we investigate the responses of the FPI with $L_1 = 20 \mu\text{m}$ and $L_2 = 50 \mu\text{m}$ coated with 150 nm $\text{Pd}_{76}/\text{Ag}_{24}$ composite film exposed to different hydrogen concentrations. The sensitivity of the sensor coated with $\text{Pd}_{76}/\text{Ag}_{24}$ composite film is comparable to the FPI with pure Pd film, while the proposed FPI sensor film exhibits good mechanical performance. The experiment reveals the potential of the FPI coated with Pd/Ag composite film for hydrogen concentration detection.

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