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Surface Plasmon Resonance Hydrogen Sensor based on Hetero-core Optical Fiber Structure

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Abstract

In this paper, we report the surface plasmon resonance (SPR) hydrogen sensor based on hetero-core optical fiber structure. This sensor consists of a multilayer stack made of a gold, a tantalum pentaoxide and a palladium (Pd) layer. It have been known that permittivity of Pd, which is a hydrogen storage metal, slightly changes with Pd hydrogenation. We experimentally obtained that SPR resonance was changed by 28 nm in wavelength with ranging from 0 to 4% hydrogen concentration. In addition, it was found that Pd thickness could tune the SPR resonance wavelength. On the other hand, its SPR broad spectrum enables the proposed sensor to be modulated with intensity-basis with an LED operation at 850 nm. It was presented that the proposed sensor induced a transmitted loss change of approximately 0.4 dB with reaction time of 40 sec. in the range of 0 – 4% hydrogen.

Keywords:Hetero-core structure; Optical fiber; Hydrogen; Palladium; Surface plasmon resonance;

1. Introduction

Recently, hydrogen has been utilized as an energy source because it is clean, sustainable and abundant energy. On the other hand, hydrogen is extremely flammable and very volatile, which makes it difficult to store and handle. A leakage of hydrogen more than 4% in air would lead to an easily ignited explosive atmosphere. Therefore, fast and reliable hydrogen sensors are desired. So far, many hydrogen sensors based on optical fiber structure have been developed [1-2]. Optical fiber sensors have estimated an very promising devices because of their ability to operate in potentially explosive environments. Most of

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optical hydrogen sensors have utilized palladium (Pd) as the sensitive material since Pd allows the selective detection of hydrogen. When Pd absorbs the hydrogen, the dielectric permittivity of Pd is changed. This change is reversible and nonlinear. Surface Plasmon Resonance (SPR) optical sensor is one of very promising sensors due to its advantages of high-sensitive, real-time and rapid detection [3]. Therefore, utilizing the SPR technique for hydrogen detection, fast and high-sensitive hydrogen sensor could be achieved. Thus, we propose the SPR hydrogen sensor based on hetero-core fiber structure consisting of a multilayer by made of a gold (Au), a tantalum pentaoxide (Ta_2O_5) and a Pd layer. Here, we discuss a shift resonant wavelength, which is tuned with the Pd thickness, based on the spectral mode operation and demonstrate the capability of the proposed sensor in the intensity operational mode for hydrogen detection.

2. Principle of method

2.1. SPR hydrogen sensor based on a hetero-core fiber structure

Fig. 1 shows the structure of a SPR hydrogen sensor based on hetero-core fiber structure. The hetero-core fiber sensor consisted of a transmission line multimode (GI) fiber and an inserted segment of a single-mode (SI) fiber, which works as a sensing element. With this structure, most of the light would leak into the cladding region of the sensing element, although it can be considered that a little light power is re-coupled to the core of multimode fiber. The leaked light into the cladding region induces evanescent interaction with surrounding properties at a condition where total reflection is satisfied. Therefore, by coating the cladding surface with a thin metal film, SPR optical arrangement could be realized similar to the kretschmann configuration sensor. The proposed hetero-core fiber SPR hydrogen sensor, which consists of a multilayer by made of Au, Ta_2O_5 and Pd layer, is shown in Fig. 1. Au is chosen since it is well known as a good candidate for SPR sensors. Ta_2O_5 tunes the SPR resonance wavelength to higher wavelength and Pd is used as a sensitive substrate for hydrogen detection.

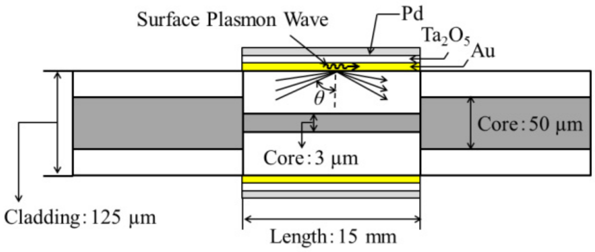

Fig. 1. Structure of SPR hydrogen sensor based on hetero-core optical fiber.

2.2. Simulation for SPR hydrogen sensor

Fig. 2 shows theoretical SPR spectra based on multi-layered model [4] for different thickness of Pd for 0 and 4% hydrogen, assuming that there would be a multi-mode distribution [5] in the hetero-core region with the corresponding incident angles in the hetero-core structure. The effects of the absorption of hydrogen on the complex permittivity of Pd can be represented by the following empirical equation:

$$\varepsilon_{pd}(c) = h(c) \times \varepsilon_{pd}(0) \quad (1)$$

Where ε_{Pd} is the complex permittivity of the pure Pd layer [6]. h(c) is nonlinear function decreasing with hydrogen con centration c% and taking values less than 1. At normal conditions, h(0%) =1.0, h(4%) =0.8.

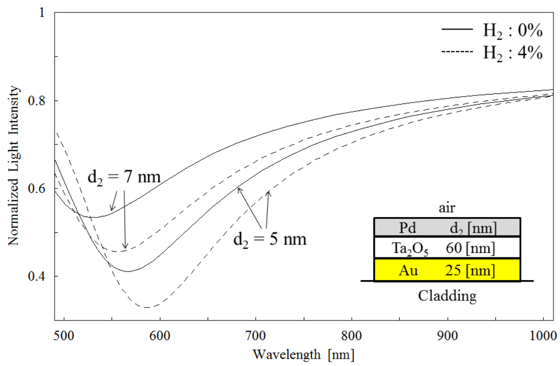

Fig. 2. Theoretical SPR spectra for 100% nitrogen and for 4% hydrogen for different thickness of Pd.

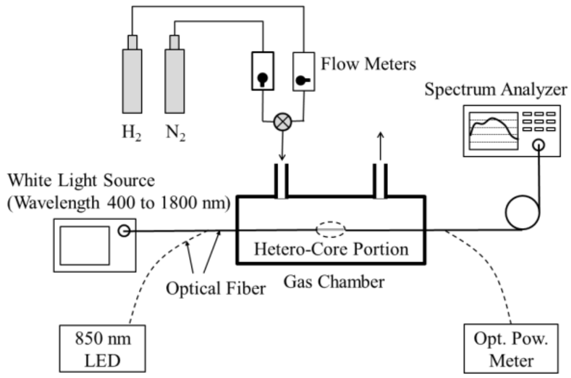

Fig. 3. Experimental setup to measure the transmitted light spectrum and intensity.

As seen in Fig. 2, the sensor responses show the spectral resonant peak of the for 0 and 4% hydrogen at the specific wavelength. Upon hydrogenation, the resonant wavelength shifts to higher wavelengths. The observed peak corresponds to the excitation of the SPR on the multi-layer film. As shown in Fig. 2, increasing the Pd thickness widens the resonance peak and increases the peak minimum value. It is worth noting that an increasing Pd thickness shifts the SPR peak toward shorter wavelengths because of the decrease of the effective index of the SP.

3. Experimental set-up

Sensors used for the experiment were fabricated with a 15-mm-long segment of a SI SMF inserted into a GI MMF fiber. Both fibers have the same cladding diameter of 125 μm, but core diameters of 3 and 50 μm, respectively. The fabricated hetero-core region was then cylindrically coated with Pd-coated with thickness of 5 and 7 nm over a 60-nm thickness Ta_2O_5 of second layer and a 25-nm thickness Au of first layer by using an RF sputtering machine (CFS-4DS-231, Shibaura Mechatronics Corp.). In order to survey the characteristics of Pd thickness, we set up the experiment using halogen lamp, whose wavelength was in the range of 400-1800 nm, and an optical spectrum analyser, as shown in Fig. 3. The sensor was placed in an acrylic gas chamber. Nitrogen was used as a carrier gas in the chamber. The flow rate of nitrogen and hydrogen, which was individually controlled with flow meters, was 1000 ml / l, respectively.

4. Results and discussion

4.1. Spectral mode operation

Fig. 4 shows SPR spectra experimentally obtained in the case of 100% nitrogen and 4% hydrogen for the sensors having the Pd thickness of 5 and 7 nm, respectively. These spectra are normalized with the air spectrum obtained by Au 25-nm thickness. For each sensors, the typical SPR curves are appeared at specific resonant wavelength for the corresponding 100% nitrogen and 4% hydrogen. Comparing the experimental results with the theoretical SPR spectra shown in Fig. 2, we can find the wavelength shifts similar to them with the increase of Pd thickness, although they appears somewhat broader than that of theoretical SPR spectra because of multi-mode power distribution with corresponding incident angles. In this work, the resonant wavelength is shifted 28 nm and 17 nm at concentration of 4% hydrogen in

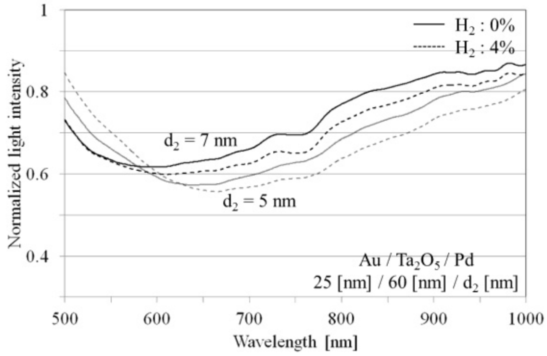

Fig. 4. Experimental results of SPR spectra for 100% nitrogen and for 4% hydrogen for different thickness of Pd. The SPR spectra normalized with air spectra obtained by Au 25-nm layer.

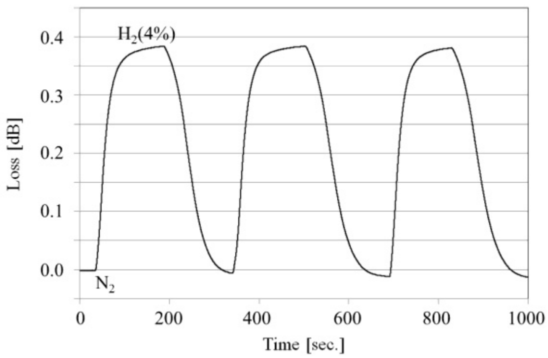

Fig. 5. Real time response in optical loss for 100% nitrogen and for 4% hydrogen concentration. Given thickness for Au, Ta_2O_5 and Pd are 25, 60, 5 nm, respectively.

nitrogen for case of the Au 25 nm / $Ta_2O_5$60 nm/ Pd 5 nm and he Au 25 nm / Ta_2O_5 60 nm/ Pd 7 nm, respectively.

4.2. Intensity mode operation

For the proposed sensor, it allows us to modulate with intensity-basis with a LED operation at 850 nm because of its SPR is broad spectrum. An additional experiment was carried out by using a LED whose wavelength is 850 nm as a light source. The experimental arrangement is shown in Fig. 3. The prepared sensor was coated with 25/60/5 (Au/ Ta_2O_5/ Pd). In Fig. 5, we have found that the proposed sensor induced a transmitted loss change of approximately 0.4 dB with time response (the time required for the sensor to reach from 10% to 90% of optical loss change) of 40 sec. in the range of 0 – 4% hydrogen. As a result, it is indicated that the proposed sensor reproductively works for 4% hydrogen with good sensitivity.

5. Conclusions

In this paper, we proposed a SPR hydrogen sensor based on hetero-core fiber structure, which is a multilayer by made of a Au, a Ta_2O_5 and a Pd layer. Experimentally obtained SPR spectra have revealed that the tested fiber sensor is capable of the spectral and the intensity mode operation by controlling the Pd layer thickness. From the experiments based on spectral mode, we obtained the typical SPR spectra for 0 and 4% hydrogen at a specific wavelength. In addition, it is observed that the presence of 4% hydrogen shifts the resonance wavelength by 28nm. In the intensity mode using the LED operating at 850 nm, we could confirm that a response time is 40 sec. and good sensitivity suitable for 4% hydrogen.

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