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Study on Sensitivity Improving of Fiber Bragg Grating Based pH Sensor

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Abstract: The key factor of the sensitivity in the FBG-based pH sensor is analyzed in detail. A multi-thin-layer structure of the gel coated cover was proposed and implemented with a special process. The sensors with the coated thickness of 420 μm, 500 μm, and 580 μm were built up, respectively. The corresponding spectral shifts of 0.08 nm, 0.13 nm, and 0.22 nm were detected when the pH sensors were soaked in the pH value of 3–9. Meanwhile, the sensor with the gel layer thickness of 580 μm was measured in the optimum measurement time period with the pH value changing from 3–12, in which the detected sensitivity of 52 pm/pH was achieved in the pH range of 6–12.

Keywords: Fiber Bragg grating, hydrogel, pH sensor, sensitivity

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1. Introduction

With advantages of high sensitivity, wavelength encoding and reflective measurement, etc. [1], fiber Bragg gratings (FBGs) have been proved to be high performance sensors in detecting physical quantities, such as the strain, temperature, displacement, acceleration. However, the FBG sensors designed for chemical detection are still in the stage of exploration, and the related studies are becoming the current research hotspots of the FBG.

Recently, a new kind of chemo-mechanical-optical sensors based on the FBG has been proposed and studied for the measurement of one of the most important chemical parameters — the pH value of solution, in which the expanding or contracting in the volume of pH sensitive hydrogel coated on the FBG will result in shifts of the reflection spectrum of the FBG. Initially, I. Yulianti [2–3] proposed and studied the FBG-based pH sensor by using two aluminum disks attached at the end of the sensing region to prevent the axial expansion of pH-sensitive hydrogel coated on the FBG. In this design, the structural complexity is introduced, and the measurement range is limited for the poor acid and alkali resistance of aluminum disks. After that, B. N. Shivananju [4] studied pH sensing by the single layer hydrogel coated FBG to obtain a sensor with the simple structure and large measurement range. However, the sensitivity of the sensor with single

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layer hydrogel coated is just 3 pm/pH. At present, FBGs-based pH sensors have not far reached a practical application level as there are some rough edges to be improved, among which, one of the important issues is to increase the detected sensitivity in the sensor.

In this paper, aiming at improving the sensitivity of FBG-based pH sensors with the simple gel layer structure, the principle of high sensitivity in the sensor is analyzed in detail, and the variational trend of the sensitivity in the FBGs pH sensor is discussed. Further, a multi-thin-layer structure of pH-sensitive hydrogel of the FBGs-based pH sensor is proposed and implemented, in which many thin pH-sensitive hydrogel layers are coated on the FBG with an improved coating process. As a result, the measuring sensitivity of the FBGs-based pH sensor is greatly improved within the range of the pH value 6–12. The principles of the FBG-based pH sensor with the high sensitivity and building up of high sensitive pH sensors, as well as the test schemes are given in Section 2. The corresponding test results and the related discussion are expressed in Section 3, and then the conclusion is given in last section.

2. Experimental measurement schemes

2.1 Principles of FBG-based pH sensor with high sensitivity

In FBG-based pH sensors, the expansion or contraction of the pH sensitive gel, which is coated on the FBG, will apply a stress along the fiber grating, resulting in a change in the grating period in the FBG, sequentially and a change in the reflected wavelength (λ_B), which is called the Bragg wavelength and can be expressed as

$$\lambda_B = 2n_{\text{eff}}\Lambda \tag{1}$$

where n_{eff} is the effective refractive index of the fiber, which is a constant for certain fiber, while Λ is the grating period. In a word, it is the stress in the pH sensitive gel layer coated on the FBG that causes the Bragg wavelength shift, in the case of the temperature of the sensor being kept constant.

Therefore, the Bragg wavelength can be expressed as following (1)

$$\frac{\Delta\lambda_B}{\lambda_B} = K_\varepsilon \cdot \varepsilon \tag{2}$$

where ε is the axial stress of the FBG, which is caused by the expansion or contraction of the pH sensitive gel layer coated on the FBG, and K_ε is a constant.

The mechanical expansion mechanism in different layers of FBGs structure was studied by B. Sutapun [5], in which, the Pd coated layer on the FBG was used to sense H_2 via the elastooptic effect. In the study, the stress applied on the FBG is given in the following equation:

$$\varepsilon_F = \delta\frac{\left(b^2 - a^2\right)Y_P}{a^2Y_F + (b^2 - a^2)Y_P} \tag{3}$$

where a and b are the radii of the FBG fiber (core and cladding) and the coated Pd layer on the FBG, respectively, while Y_F and Y_P are Young moduluses of the fiber and Pd, and δ is the stress coefficient. As Y_F=73 GPa [6], Y_P=128 GPa [7], the applied stresses on the FBG versus different coated layer thicknesses are given in Fig. 1(a), in which, the stresses hardly increase when b is beyond 250 μm.

In our case, however, things are totally different. Instead, the FBG is coated with the gel layer whose Young modulus, denoted as Y_H, is much smaller than that of the fiber (Y_F is in the order of dozens GPa, and Y_H is only in the order of less than MPa [8]), so the axial stress in the FBG core can be simplified as

$$\varepsilon_F \approx \delta\frac{(b^2 - a^2)Y_H}{a^2Y_F} \tag{4}$$

where δ is the stress coefficient proportioning to the pH value, and the related curve of ε_F versus the coated radius b is shown as Fig. 1(b). It is observed from Fig. 1(b) and (4) that: firstly, ε_F/δ is much smaller in comparison with the case of the FBG coated with a metal layer like Pd; secondly, ε_F/δ is quite little when b is small. As the thickness of the gel layer coated on FBGs in the present pH FBG based sensors is quite small, the measuring sensitivity is very low. It is obvious that ε_F/δ will be greater when b increases, which will result in a larger Bragg wavelength shift, that is to say, a high sensitivity of the sensor can be achieved.

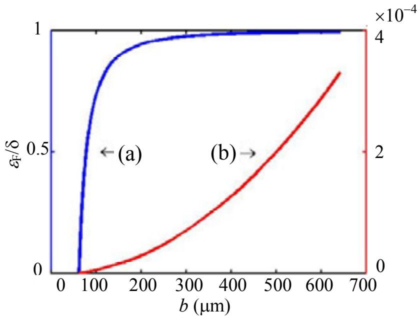

Fig. 1 Comparision between the Pd and the hydrogel coated layers: (a) stress on the FBG versus different radii b of the Pd coated layer and (b) stress variation trend versus different radii b of the hydrogel layer coated on the FBG.

2.2 Building up of high sensitive FBG pH sensor and test schemes

From the above analysis, it is clear that a higher sensitivity in the FBG-based pH sensor can be achieved by the way of getting a thicker gel layer coated on FBGs, in which the cylindrical symmetry along the fiber axis needs to be ensued. The symmetrical thickness of the gel layer, however, is not the only issue to be considered. Besides, the response time and tensile strength of the FBG sensor are the other factors needed to be taken into account. Therefore, in practical design, instead of purchasing a big radius in the gel layer, a moderate radius will be more reasonable for a high performance of the FBG-based pH sensor.

The hydrogel used to be coated on the FBG is a 2-hydroxyethyl methacrylate(HEMA)-acrylic acid (AA) comonomer gel sensitive to variations in the environmental pH [9]. As the pH-sensitive hydrogel used is quite good at liquidity, which is hard to be coated on the fiber grating, a multi-thin-layer structure of the pH sensor is designed as shown in Fig. 2, where the so-called multi-thin-layer structure is built up via our unique coating-curing process, in which the coated thin thickness and the curing time need to be precisely controlled in order to make sure of the symmetry of the coated layer and the stick strength between layers.

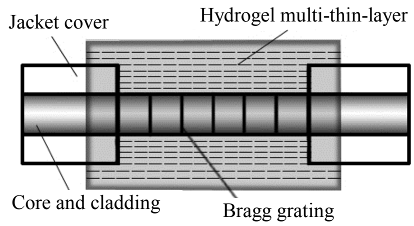

Fig. 2 Schematic diagram of a multi-thin-layer structure of the FBG-based pH sensor.

In our scheme, the gel layer thicknesses of 420 μm, 500 μm, and 580 μm were designed and built up for the experiment. The test setup is shown as Fig. 3, where an amplified spontaneous emission (ASE) was used as the broadband light source, which was coupled into the FBG pH sensor with the 2*2 coupler. During the tests, the different Bragg wavelength shifts in the FBG pH sensor, in different time periods, were detected by the optical spectrum analyzer (OSA), corresponding to being soaked in different pH values of the solution, whose range was from 3.0 to 12.0 of the pH value.

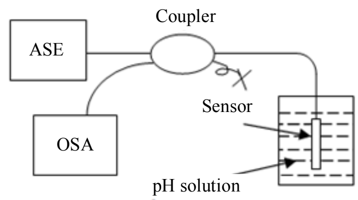

Fig. 3 Experimental test scheme for the FBG-based pH sensor.

Two measurement schemes have been carried out in our experiments, among which, the Bragg wavelength shifts in three FBG-based pH sensors with the gel layer thicknesses of 420 μm, 500 μm, and 580 μm versus different pH solutions and the Bragg wavelength shift of the pH sensor with a gel layer thickness of 580 μm versus different pH solutions in the optimum measurement time period, were tested.

3. Experimental result and discussion

3.1 Test results of pH sensors with different coated thicknesses

The test reflected spectral shifts of the FBG-based pH sensors we prepared with different coated thicknesses, corresponding to the gel layer thicknesses of 420 μm, 500 μm, and 580 μm, respectively, are shown in Fig. 4. It can be seen from the figure that the maximum wavelength shifts in the reflected spectra of the pH sensors were 0.08 nm, 0.13 nm, 0.22 nm, respectively, corresponding to the gel layer thicknesses of 420 μm, 500 μm, and 580 μm, when the pH value of the solution changed from 3.0 to 9.0. It is obvious that the thicker the gel layer is, the greater the stress acting on the FBG will be, via the expansion of the hydrogel, as a result, the maximum shift of the Bragg wavelength is greater, which means a higher test sensitivity in the pH sensors. Besides, Fig. 4 suggests that the high sensitive response of the three sensors is main in the pH range of 6.0–9.0. The average sensitivities in this test range were worked out with the result of 15 pm/pH, 28 pm/pH, and 53 pm/pH, which corresponded to the gel layer thicknesses of 420 μm, 500 μm, and 580 μm, respectively. Comparing with the sensitivity of 3 pm/pH got with using the single hydrogel coated FBG in the previous research [4], the sensitivity in our case was improved by an order of magnitude. The test results showed that the FBG-based pH sensor with the thicker gel layer had higher sensitivity, which was consistent with the above analysis. Meanwhile, the pH sensor with a gel coated layer thickness of 580 μm seems to have more practical value with higher sensitivity.

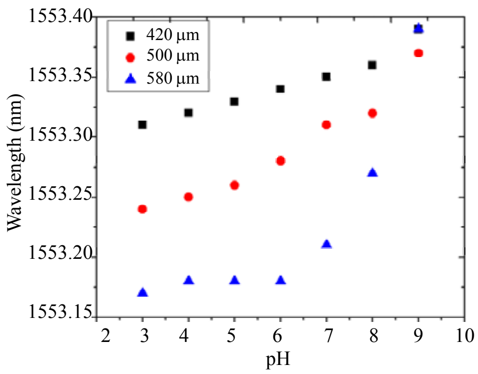

Fig. 4 Spectral shifts of FBG-based pH sensors versus different coating thicknesses (the Bragg wavelengths in the free state are 1553.35 nm, 1553.31 nm, and 1553.21 nm, respectively).

3.2 Test results of pH sensors in the optimum measurement time period

An additional study was undertaken to optimize the sensing performance of FBG-based pH sensors via the optimum measurement time. The affect of the polymerization time of pH-sensitive hydrogel on the elastic modulus was studied by B. D. Johnson [9], in which the Young modulus of hydrogel had different values in different test time periods, and the maximum and stable value was in the test time period of 4 min. It suggests that 4 min is the optimum measurement time and is a key factor in optimizing the sensing performance of the FBG-based pH sensors when (4) is also taken into consideration. Thus, the sensing performance of the pH sensor with a gel layer thickness of 580 μm versus different pH solutions in the optimum measurement time period of 4 min was tested. In the test experiment, the Bragg wavelength of the FBG was 1544.26 nm in the free state, the bandwidth of it was 3 dB, and the reflectivity of the grating was over 90% by the ultraviolet(UV)-writing technology [10]. The values of the Bragg wavelength were determined by the peak wavelength tracing through setting the resolution of the optical spectrum analyzer (Yokogawa AQ6370B) to be 0.01 nm.

The test Bragg wavelength shifts of the FBG-based pH sensor with the gel layer of 580 μm versus different pH values in the range from 3.0 to 12.0 in the measurement time period of 4 min, are shown in Figs. 5 and 6, in which Fig. 5 gives the different reflection spectra of the FBG-based sensor corresponding to different pH values, showing that the Bragg wavelength experiences a red shift as the pH value increases. From Fig. 6, it can be seen that the Bragg wavelength shift is proportional to the changes in the pH value in the range of 6–12. The sensitivity in the pH range of 6–12 was worked out with the result of 52 pm/pH by using the linear fitting. Additionally, the Bragg wavelength observed in the experiment was a stable value in the time period from 3 min to 5 min, which was in good agreement with the result of B. D. Johnson's study [9]. The study result showed that an FBG-based pH sensor with practical sensing performance could be got, by optimizing with the gel coated layer of 580 μm and optimum measurement time of 4 min, of which the measurement range was 6 pH–12 pH value, and the sensitivity was 52 pm/pH.

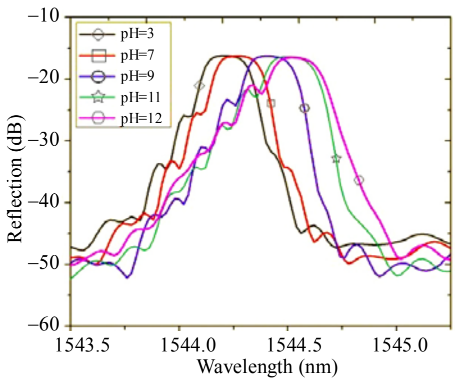

Fig. 5 Reflected spectra of the FBG-based pH sensor measuring in different pH values.

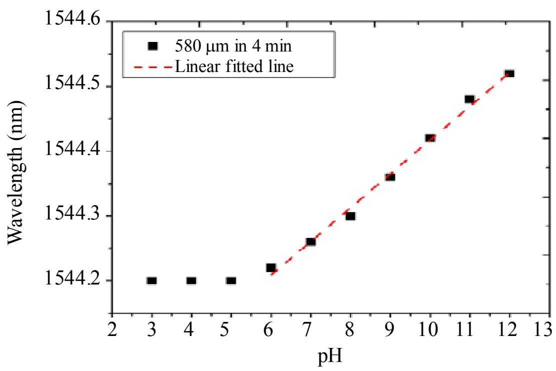

Fig. 6 Spectral shifts of the FBG-based pH sensor with the coated thickness of 580 μm in 4 min.

4. Conclusions

With regard to increasing the sensitivity of present pH FBG sensors, the related factors in determining the sensitivity of the pH sensors are analyzed in detail. The stress, resulted from the expansion or contraction of the pH sensitive gel coated on FBGs, versus the thickness of a coated layer is given in curve, which indicates that a higher sensitivity of the FBG-based pH sensor is achieved via increasing the radius of the gel layer coated on FBGs, with a cylindrically symmetrical structure . A multi-thin-layer structure of the gel coated cover was proposed in order to ensure the cylindrical symmetry along the fiber axis when the coated layer was getting thicker, which was implemented via our special "coating over curing" process. With this design and the process, three kinds of FBG-based pH sensors were built up, with different coating thicknesses of 420 μm, 500 μm, and 580 μm, respectively. The corresponding spectral shifts of 0.08 nm, 0.13 nm, and 0.22 nm were detected when the pH sensors were soaked in the pH value of 3–9. Besides, the related spectral shifts were also measured in the optimum responding time period of 4 min for the thickness of 580 μm pH-sensitive gel layer with the pH value changing from 3–12. The test results with the good performance were realized by optimizing the sensor with the optimum measurement time. Yet, it needs to be mentioned that in the acid pH value lower than 6, the design in this paper did not work, which will be the next job to be focused on. Anyway, the analysis, the design, and the experiments done in this paper will provide a good reference for the practical pH sensor design.

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