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Wide-range pH sensor based on a smart-hydrogel-coated long-period fiber grating

Satyendra Kumar Mishra, Bing Zou, and Kin Seng Chiang

Abstract—We report a fiber-optic sensor for pH measurement, which is a long-period fiber grating (LPFG) coated with smart hydrogel. When the pH of a solution that surrounds the hydrogel coating changes, the volume of the coating and hence its refractive index are changed, which results in a shift in the resonance wavelength of the LPFG. Our experimental sensor shows a sensitivity of ~ 0.66 nm/pH over the pH range from 2 to 12 and a response time of less than 2 seconds. The effects of the refractive index of the surrounding solution and temperature variations on the sensor are also measured.

Index Terms—Chemical sensors, fiber gratings, optical fiber sensors.

I. INTRODUCTION

Estimation, monitoring, and control of the pH of a solution are required in a wide range of chemical, biological, and environmental applications, such as titration, blood sampling, water-quality monitoring, etc. [1], [2]. Most of the optical pH sensors are based on the use of a coating material with a pH-sensitive optical property. Indicator dyes [3] and hydrogels [4] are the most popular coating materials used in optical pH sensors, as the absorbance or fluorescence of the dyes and the volumes of the hydrogels are sensitive to the pH change. In general, hydrogel-coated sensors can operate over a wider range of pH values and are more stable, compared with those using dyes. Hydrogels used for pH sensing contain around 30% of water by weight and are hydrophilic polymers in nature. A change in the pH leads to swelling or shrinkage of the hydrogel, which thus causes a change in its volume and hence its refractive index [5]–[9]. A number of sensing structures based on pH-sensitive hydrogel have been reported, such as micromechanical cantilever [10], micro gravimetric transducer [11], gold nanocrescent array [12], reflective diffraction grating [13], and fiber-based structures [14]. Among these structures, the fiber-based sensors exhibit several distinct advantages: immunity to electromagnetic interference, small sensing probe (important for costly sample and small sample quantity), and remote sensing ability [15]–[17].

Several fiber structures utilizing pH-sensitive hydrogel are available, which include fiber interferometer [18], surface plasmon resonance (SPR) fiber structure [19], fiber Bragg grating (FBG) [20], and long-period fiber grating (LPFG) [21].

LPFG-based pH sensors have a leading edge for their easy fabrication and high sensitivity. An LPFG has a periodic modulation of the refractive index along the fiber with a typical period in the range of 100 μm to 1 mm. It acts to couple light from the core mode to the co-propagating cladding modes of the fiber, which results in a transmission spectrum containing a series of attenuation bands centered at discrete resonance wavelengths. Each attenuation band corresponds to the coupling to a different cladding mode. LPFGs have found wide applications as sensors owing to their sensitivity to the surrounding medium [22], [23]. In a hydrogel-coated LPFG pH sensor, a change in the pH of the surrounding medium can change the refractive index of the hydrogel coating, and consequently shift the resonance wavelengths of the LPFG.

Most of the hydrogel-coated fiber sensors demonstrated operates over only limited pH ranges. For example, the interferometric sensor [18], the FBG sensor [20], and the LPFG sensor [21] work for the pH ranges 2.5 – 6.5, 1 – 7, and 6 – 8, respectively. The reason is that the hydrogels used in these sensors are synthesized with poly(vinyl alcohol) and poly(acrylic acid), which do not show any response at large pH values [24]. In this paper, we demonstrate a hydrogel-coated LPFG sensor that can measure almost the full range of pH.

The hydrogel used in our sensor is a “smart hydrogel” [25], which is mainly synthesized with acrylamide, bisacrylamide solutions, and methacrylic acid. A change in the pH of the solution in contact with the hydrogel changes the amount of the dissociated carboxylic ions in the hydrogel, which leads to a change in the volume and hence the refractive index of the hydrogel. The pH response of the hydrogel depends critically on the relative concentrations of acrylamide, bisacrylamide solutions, and methacrylic acid used in the synthesis process. By using a sufficient amount of methacrylic acid, it should be possible to extend the pH range that the hydrogel can measure [26]. A hydrogel prepared this way has been utilized in an SPR fiber sensor [19] to achieve a combined pH range of 3 – 6 and 8 – 11. Our carefully synthesized hydrogel used in the present study can work for a much wider pH range.

The LPFG used in our study was fabricated in a single-mode fiber by a CO₂-laser writing method. With the LPFG coated with a thin layer of smart hydrogel, the shift in the resonance wavelength provided a measure of the pH of the solution in contact with the coated LPFG. Our experimental sensor shows an average sensitivity of ~ 0.66 nm/pH over the pH range from 2 to 12 (limited by the pH solutions available) and the response time of the sensor is less than 2 seconds. We also characterize the temperature sensitivity of the sensor and its response to a change in the refractive index of the surrounding solution.

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II. SENSOR PREPARATION AND EXPERIMENTS

A. Grating fabrication

An LPFG formed in a single-mode fiber can be designed to couple the core mode to a co-propagating cladding mode of the fiber. The resonance wavelength, at which the coupling effect is strongest, is determined by the phase-matching condition:

$$\lambda_0 = (n_{core} - n_{clad})\Lambda \quad (1)$$

where Λ is the grating pitch and n_{core} and n_{clad} are the effective indices of the core mode and the cladding mode, respectively. As the resonance wavelength of an LPFG can be changed by physical parameters, such as strain, temperature, and external refractive index, LPFGs have been proposed as sensors for various applications [22], [23]. The LPFG used in our study was written in a germanium-boron co-doped single-mode fiber (Fibercore, PS 1250/1500) with a CO₂ laser (CO₂-H10, Han's Laser), which had a maximum average output power of 10 W at 10.6 μm [27]. The frequency of the laser pulses was fixed at 5 kHz and the average output power was set at 0.75 W. The laser beam was focused onto the fiber (with its jacket removed) to a spot of $\sim 90 \mu\text{m}$ and controlled by a computer to scan point-wisely across the fiber. The laser beam was advanced along the fiber at steps to repeat the transverse scanning process with each step equal to the grating period. The LPFG written had a period of 330 μm and a length of 100 periods. The CO₂-laser energy density used was 3.5 J/mm². The measured transmission spectrum of the LPFG is shown in Fig. 1, where the resonance dip at $\sim 1556 \text{ nm}$ is caused by the coupling from the core mode to the LP₀₈ cladding mode.

B. Hydrogel synthesis and coating preparation

The chemicals for synthesizing the smart hydrogel used in our pH sensor are N-tetramethylethylenediamine (TEMED), acrylamide, bisacrylamide, ammonium persulphate (APS), methacrylic acid, hydrochloric acid, methacrylic acid, hydrochloric acid, ethanol, glacial acetic acid, sodium hydroxide, 3-trimethoxysilylpropylmethacrylate, and 3-trimethoxysilyl propyl methacrylate. All these chemicals were purchased from J&K Pvt. Ltd. Acrylamide/bisacrylamide, TEMED, and APS were used as cross-linking reagent, catalyst, and initiator, respectively, in the synthesis process.

To synthesize the pH-sensitive hydrogel, we first prepared aqueous solutions of acrylamide and N, N-methylene

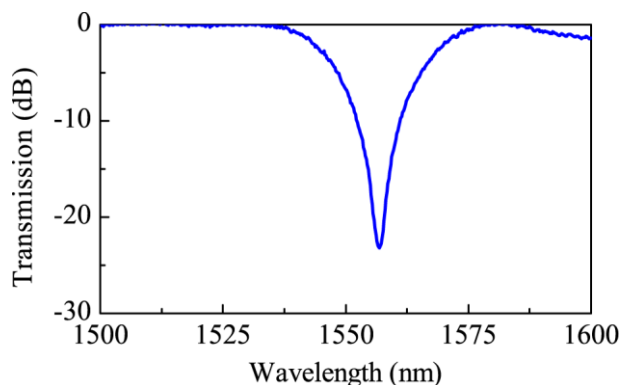


Fig. 1. Measured transmission spectrum of the LPFG (in air) used in our pH sensor.

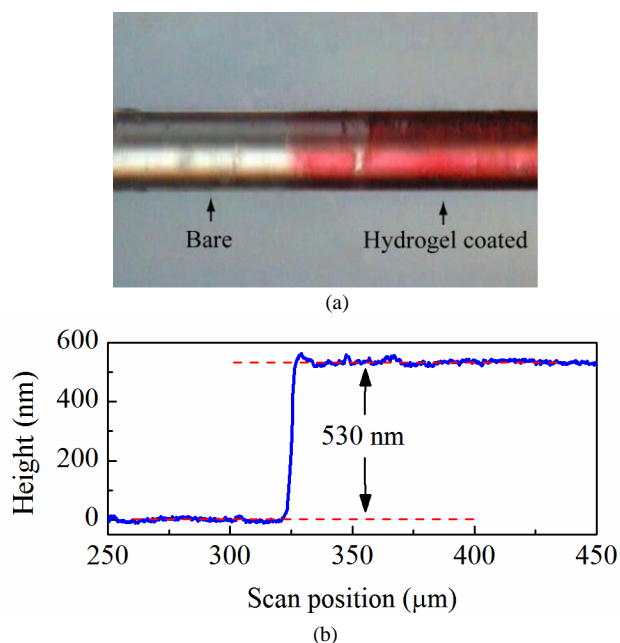


Fig. 2. (a) Microscopic image of a hydrogel-coated fiber and (b) the height profile across the bare section and the coated section of the fiber, showing a coating thickness of $\sim 530 \text{ nm}$.

bisacrylamide (19:1). We next prepared a polymerization medium by mixing 5.4 ml Millipore water, 800 μl acrylamide/bisacrylamide solutions (40%), and 400 μl methacrylic acid. We then added 10 mg ml⁻¹ concentration of ammonium persulphate to the polymerized medium as a chemical initiator and mixed the solution with TEMED to catalyze the reaction. Polymerization of the catalyzed solution was initiated upon exposure to light and it took 15 hours to complete the process and produce the hydrogel.

The fabricated LPFG was dipped into the hydrogel for 5 minutes and the coated LPFG was kept overnight for drying. Figure 2(a) shows a microscopic image of a fiber coated with hydrogel. As the hydrogel coating was transparent, we dipped the coated section into red ink and then removed part of the coating with a razor blade to make the coating visible. The thickness of the hydrogel coating, measured with a step profiler (Ambios Technology, Model XP-2), was $\sim 530 \text{ nm}$, as shown in Fig. 2(b). With the hydrogel coating, the resonance wavelength of the LPFG shifted towards the shorter wavelength by a few nanometers.

We further prepared solutions with different pH values for characterizing the pH sensor by mixing Millipore water, NaOH, and HCl in proper proportions. The pH values of these solutions were measured with a commercial pH meter (HANNA, HI98128 pHep5). Eight such aqueous solutions were prepared, with pH values varying from 2 to 12. The refractive indices of these solutions were all around 1.33.

C. Experimental setup

Figure 3 shows the experimental setup for the pH measurement with the hydrogel-coated LPFG. Light from a broadband source (Amonics, ALS-15CL) was launched into the LPFG at one end and detected with an optical spectrum analyzer (Yokogawa, AQ6370) at the other end. The sensor was sealed in a flow cell, which contained a solution with

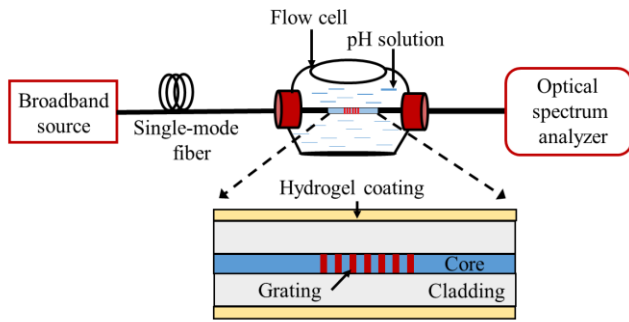


Fig. 3. Schematic diagram of the experimental setup.

known pH. The transmission spectra of the sensor were recorded for solutions with different known pH values.

III. RESULTS AND DISCUSSIONS

A. pH sensing

Figure 4 shows the transmission spectra of the pH sensor for pH values varying from 2 to 12 at 22 °C. In principle, the sensor could operate at even lower or higher pH, but such extreme applications are rare. To minimize the hazard in preparing solutions with extreme pH, we limited the pH range from 2 to 12, which is already the widest range ever reported for fiber-based pH sensors. As shown in Fig. 4, the resonance wavelength of the LPFG increases with the pH value. When the pH value increases, more carboxylic ions in the hydrogel are dissociated. This leads to more electrostatic repulsion among the ionized carboxylic groups and, consequently, a swelling of the hydrogel and more effective absorption of water by the hydrogel. The hydrogel has a refractive index of ~ 1.423 , which is much larger than that of water (~ 1.33). Therefore, the absorption of water from the standard pH solution prepared by the hydrogel coating lowers the refractive index of the coating. As the core mode of the fiber is well confined in the core area, its effective index does not change with the refractive index of the hydrogel coating. On the other hand, the effective index of the cladding mode decreases with the refractive index of the hydrogel coating and hence with an increase in the pH. According to Eq. (1), the resonance wavelength increases with a decrease in the effective index of the cladding mode and, therefore, should increase with the pH, which agrees with the experimental observation. In addition, a decrease in the refractive index of the hydrogel coating should provide a better confinement for the cladding mode and thus enhance the coupling between the core mode and the cladding mode, which explains the increase in the strength of the resonance dip as the pH increases, as shown in Fig. 4.

Figure 5 shows the variation of the resonance wavelength with the pH values of the aqueous solutions. The experimental data are fitted with a third-order polynomial curve. As the pH value increases from 2 to 12, the resonance wavelength shifts from 1543.6 nm to 1550.2 nm, which corresponds to a sensitivity of ~ 0.66 nm/pH. The uncertainties in the measured resonance wavelengths are about $\pm 0.15 - 0.3$ nm. We replaced the hydrogel-coated LPFG with a bare LPFG and repeated the measurements. We measured the resonance wavelength of the

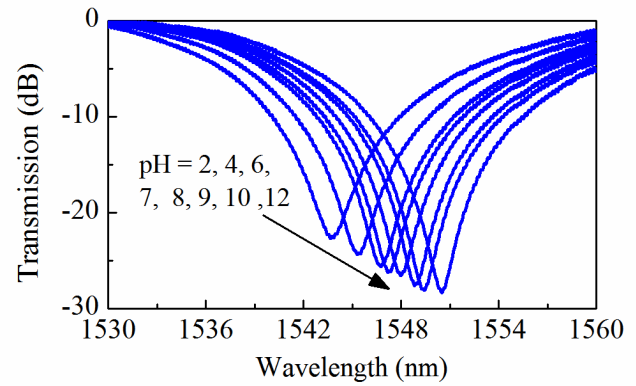


Fig. 4. Transmission spectra of the hydrogel-coated LPFG measured for different pH values.

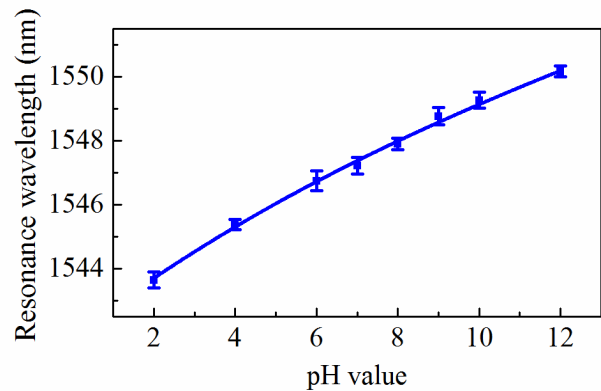


Fig. 5. Variation of the resonance wavelength with the pH value of the aqueous solution for the hydrogel-coated LPFG.

bare LPFG over a period of one hour in the same laboratory environment and obtained an uncertainty of ~ 0.24 nm, which is close to that obtained for the hydrogel-coated LPFG. We believe that the uncertainties were mainly due to temperature variations and other environmental disturbances during the measurements. The hydrogel-coated LPFG produced stable and repeatable results.

To measure the response time of the pH sensor, laser light at a wavelength of 1540 nm (Agilent 8164B) was launched into the sensor and measured with a photo-detector. The output from the photo-detector was recorded with an oscilloscope at a

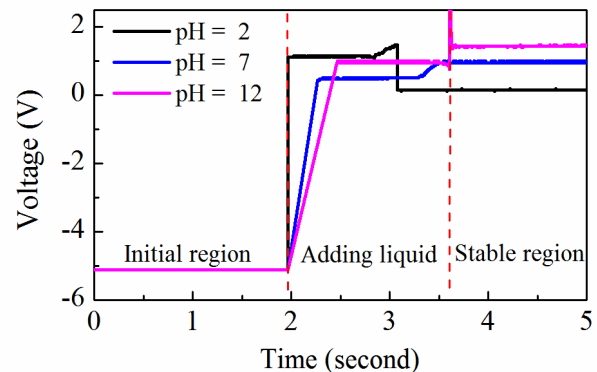


Fig. 6. Temporal response of the hydrogel-coated LPFG pH sensor measured at the wavelength 1540 nm.

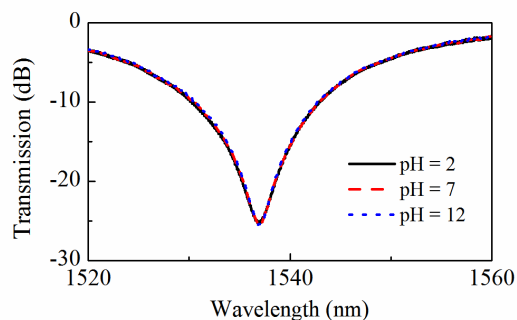


Fig. 7. Transmission spectra of a bare LPFG measured for different pH values.

sampling interval of 0.02 second. A typical oscilloscope display of the measurements is shown in Fig. 6. When the prepared solution comes in contact with the hydrogel, the ionization process in the hydrogel starts to take place. After some initial fluctuations, the sensor output becomes stable. As shown in Fig. 6, the response time of the sensor is less than 2 seconds, which is short, compared with many other hydrogel-based pH sensors [4].

To confirm that the shift in the resonance wavelength of the hydrogel-coated LPFG shown in Fig. 4 is not due to a change in the refractive index of the prepared solution, we used a similar bare LPFG to probe the prepared solutions. As shown in Fig. 7, there is no significant shift in the resonance wavelength for the bare LPFG with the pH. This is expected, as all the aqueous solutions prepared have close refractive indices and their values (~ 1.33) are much lower than that of the fiber glass (~ 1.44). The results in Fig. 4 and Fig. 7 confirm that the hydrogel-coated LPFG responds to the pH of the solution instead of the refractive index of the solution.

B. Effect of the external refractive index

Because the aqueous solutions we prepared for different pH values have close refractive indices, the hydrogel-coated LPFG responds only to a variation in the pH. In general, however, the resonance wavelength of an LPFG is sensitive to the refractive index of the surrounding medium [28]. For the measurement of the pH of a solution, it is necessary to know the refractive index of the solution. In other words, the effect of the external refractive index on the resonance wavelength of the hydrogel-coated LPFG needs to be known for sensor calibration. For this purpose, we applied different index-matching liquids (Cargille Labs) to the hydrogel-coated LPFG and measured the corresponding resonance wavelengths. These liquids had the same pH value, which was close to 7. Figure 8 shows the variation of the refractive index of the liquid, i.e., the external refractive index, with the resonance wavelength for the hydrogel-coated LPFG.

C. Effect of temperature variations

We also measured the temperature dependence of the LPFG response by placing the flow cell on a hot plate. The variations of the resonance wavelengths of the LPFGs with and without the hydrogel coating are shown in Fig. 9. We used the solutions with pH = 2, 7, and 12 for the hydrogel-coated LPFG and the solution with pH = 12 for the bare LPFG. The thermal characteristics are approximately the same for both the coated

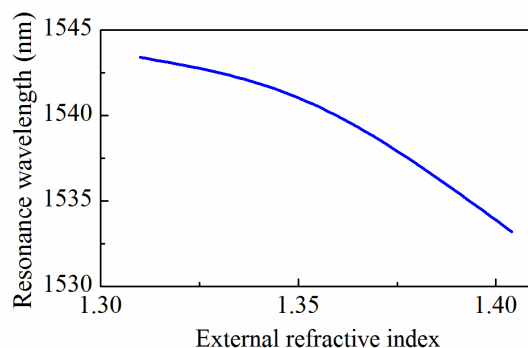


Fig. 8. Variation of the resonance wavelength of the hydrogel-coated LPFG with the external refractive index.

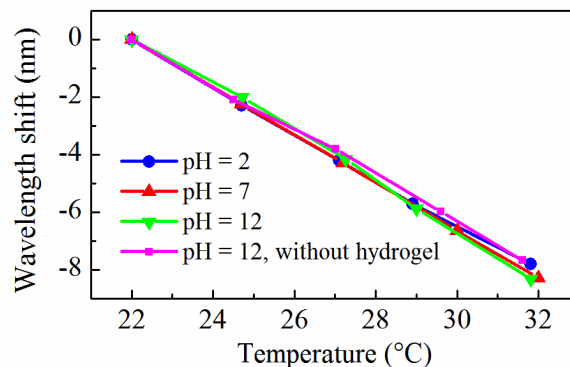


Fig. 9. Variation of the resonance wavelength of LPFG probes with the temperature. The hydrogel-coated probe was measured in solutions with pH values of 2, 7 and 12. LPFG without hydrogel was measured in solution with a pH value of 12 for reference.

and the uncoated LPFG. The wavelength shift is about 8 nm towards the shorter wavelength, when the temperature changes from 22 to 32 °C, which gives a temperature sensitivity of ~ 0.8 nm/°C. The temperature sensitivities of the bare and the coated LPFG measured in air were also close to this value, which indicates that the temperature sensitivity of the sensor is dominated by the intrinsic temperature sensitivity of the LPFG. The thin hydrogel coating and the temperature dependence of the refractive index of the aqueous solution ($\sim 10^{-4}/^\circ\text{C}$) had little influence on the temperature sensitivity of the sensor. According to our results, a fluctuation of ± 0.5 °C in the temperature can cause an uncertainty of approximately ± 0.61 in the pH (the pH sensitivity of the sensor is ~ 0.66 nm/pH), which, to a large extent, accounts for the uncertainties in our experiments. Therefore, for accurate measurements, it is necessary to calibrate away the thermal effect of the sensor. Methods are also available to reduce the temperature sensitivity of an LPFG [29], which should be applicable to our pH sensor.

IV. CONCLUSION

We have fabricated and characterized a smart-hydrogel-coated LPFG for pH measurement. The pH of the liquid in contact with the hydrogel coating can cause the coating to swell or shrink and hence change its refractive index, which gives rise in a shift in the resonance wavelength of the LPFG. Our typical experimental sensor shows a pH sensitivity of ~ 0.66 nm/pH

over the pH range from 2 to 12 and a response time shorter than 2 seconds. The pH sensitivity could be further increased by using an etched LPFG [28]. The proposed pH sensor has a number of advantages: low cost, wide sensing range, high sensitivity, fast response, small sensing area, reusability, and the capability of remote sensing, and could find applications in medicine, chemistry, biology, environmental science, food science, water treatment and purification, etc.

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