A pH Sensor Based on Corrugated Long-Period Fiber Grating

Chia-Chin Chiang, Cheung-Hwa Hsu¹,* and Chao-Hui Ou¹

Department of Mechanical Engineering, National Kaohsiung University of Applied Sciences,
415 Chien Kung Road, Kaohsiung 807, Taiwan
¹Department of Mold and Die Engineering, National Kaohsiung University of Applied Science,
415 Chien Kung Road, Kaohsiung 807, Taiwan

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In the present study, we propose a pH sensor based on a corrugated long-period fiber grating (CLPFG). CLPFG is highly sensitive to refractive index changes, and, with an appropriate sensitive coating, can detect a variety of chemical parameters. CLPFG is designed to act as a spectral loss device that couples with a discrete wavelength out of the optical fiber as a function of the surrounding refractive index. By applying a special coating that changes the refractive index through the absorption of target molecules to the CLPFG surface, the CLPFG can become a transducer for chemical measurement. In this paper, the incorporation of a polyvinyl alcohol and polycyclic acid (PVA-PAA) coating onto CLPFG to produce an optical-fiber-based pH sensor with a pH detection range from 2 to 6 is discussed.

1. Introduction

A long-period fiber grating (LPFG) has been applied in the development of optical sensing systems,¹ and has increasingly been applied in sensing for strain,¹ temperature,² and refractive indices³–⁴ and pH.⁵–⁶ LPFGs promote coupling between the propagating core and cladding modes, providing an attenuation loss band, which can be used for sensor applications. In 2009, Chiang et al.⁷ reported on the use of a sandwiched SU-8 thick photoresist technique for the fabrication of a corrugated long-period fiber grating (CLPFG) for potential applications as a sensor. CLPFG sensors possess various advantages over conventional sensors, such as small dimensions, light weight, good sensitivity, good long-term stability, corrosion resistance, and resistance to magnetic and electromagnetic interference.

*Corresponding author: e-mail: chhsu@kuas.edu.tw
The CLPFG sensor element is highly sensitive to refractive index changes and, with the appropriate coatings, can be designed to be sensitive to a variety of chemical parameters. In this study, the feasibility and performance of the sensor were investigated through the application of unique pH-sensitive hydrogel coatings to the CLPFG sensor element.

2. Materials and Methods

2.1 CLPFG refractive index calibration

The CLPFG was fabricated on dispersion-shifted fiber (DSF) using lithography and etching processes to form the grating pattern. Figure 1 shows the schematic diagram of the characterization measurement of the CLPFG. This measurement system consisted of a superluminescent laser diode (SLD), an optical spectrum analyzer (OSA), and a precise linear stage. When the force loading was applied on the CLPFG, the refractive index of the core generated a periodic distribution along the longitudinal axis of the fiber to obtain the characteristics of the CLPFG.

The CLPFG was submerged in a selected refractive index fluid, and the spectra were recorded using OSA. After each measurement was recorded, the CLPFG was cleaned thoroughly until the spectral response returned to the original state.

2.2 Coating process

Polymer gel preparation was the first step in the coating process. Ethyl acetate is primarily used as a solvent and diluent because of its low cost, low toxicity, and agreeable odor. Conversely, it is rarely selected as a reaction solvent because it is prone to hydrolysis and transesterification. It is also very volatile and has a low boiling point. Owing to these properties, it can be removed from a sample by heating the sample in a hot water bath and providing ventilation with compressed air. Thus, ethyl acetate was
used to mix polyvinyl alcohol (PVA) with polyacrylic acid (PAA); the related schematic coating process is shown in Fig. 2. The polymer gel was made by using 80% PVA and 20% PAA.

More specifically, PVA with an average molecular weight of 78000 (Polysciences, Inc.) and PAA with an average molecular weight of 90000 were dissolved in ethyl acetate solvent in relative proportions of 80 and 20% by weight, respectively. A CLPFG was first vertically inserted into, then vertically removed from this polymer solution at a speed of 10 mm/s. After that, the CLPFG was dried using a heater at 80 °C for 20 min. Next, the coating of the CLPFG was thermally crosslinked at 130 °C for 60 min. After the coating, the CLPFG sensor was allowed to soak for 24 h in buffer solutions with pH ranging from 2 to 8. Finally, the sensor measured the pH of the buffer solution.

3. Results and Discussion

The CLPFG exhibits the resonant dip spectrum for a particular wavelength. When CLPFG is subjected to mechanical forces or external environmental change, such as changes in refractive index or temperature, the transmission spectra are changed through wavelength shift or resonance attenuation loss. Figure 3 shows the characterization of CLPFGs with various force loadings. The transmission loss of the resonant dip in the spectrum changes with the loading. Furthermore, when there is no loading, no resonant dip will be observed. Consequently, the wavelength variation is a better candidate for the pH sensing.

For the refractive index calibration test, the CLPFG was submerged in the refractive index standards. There are no data points above 1.5 because the CLPFG spectral dip would disappear above this refractive index. This is due to the characteristics of the long-period grating. Figure 4 shows a plot of the spectral loss dips that occur as a result of immersing the CLPFG in the various refractive index standards. The resolution of the OSA used for this test was 0.2 nm. By tracking the spectral loss dip, the CLPFG can be used to measure refractive index changes in the surrounding medium.

![Fig. 2. Schematic of the coating process.](image-url)
As shown in Fig. 5, when the refractive index was increased, the wavelength decreased. The CLPFG response had its highest sensitivity to the refractive index changes between 1.4 and 1.5. This information is critical for developing sensors with increased sensitivity to a desired environmental parameter. When the refractive index of the solution is above 1.5, it will be too close to the refractive index of the fiber cladding and the spectral loss dip will disappear.

To test its ability to sense pH, the CLPFG coated with PVA-PAA was immersed in the various buffer solutions with pH levels ranging from 2 to 8, and the resultant changes in the spectral response were observed. As shown in Fig. 6, the resonant dip
wavelength of the pH sensor shifted with various pH levels. Figure 7 shows a plot of pH versus wavelength from the spectra. According to this plot, the wavelength shift of the pH sensor was sensitive between pHs 2 and 6. However, the wavelength shift of the pH sensor was negligible between pHs 6 and 8. The experimental results show that the resonant wavelength shifted 10.5 nm between pHs 2 and 6; accordingly, the sensitivity of the pH sensor was 2.625 nm/pH.
4. Conclusions

The optical fiber long-period grating is not only a highly sensitive refractive sensor, it can also be used to determine the pH of a solution by applying a pH-sensitive hydrogel coating. CLPFG coated with PVA-PAA demonstrated significantly sensitive measurements between pHs 2 and 6, with a sensitivity of at least 2.625 nm/pH.

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