Reflective Optical Fiber SPR Sensor for Simultaneous Measurement of Glucose Concentration and Temperature

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To achieve a compact and robust structure, a reflective optical fiber surface plasmon resonance (SPR) sensor is proposed for the simultaneous measurement of glucose concentration and temperature. The fiber probe uses the multimode optical fiber-single mode optical fiber-multimode optical fiber (MSM) heterojunction structure, in which the region with a gold film is used to sense glucose concentration, while the other region with a gold film and a polydimethylsiloxane@gold (PDMS@Au) complex is used to sense temperature. A silver film for light signal reflection is coated on the end face of the fiber probe to improve signal-to-noise ratio (SNR). The sensor successfully overcomes cross-sensitivity and achieves sensitivities of 3.05 nm/% and -1.69 nm/°C for glucose concentration and temperature, respectively. The proposed sensor can realize high-precision sensing of glucose and other biological analytes with temperature compensation. In addition, the reflective sensing structure makes the system compact and flexible, which enables remote detection and in-situ monitoring. The proposed sensor has a wide range of applications in the fields of food safety, biochemistry, and environmental monitoring.

1. Introduction

Optical fiber SPR sensors are prepared by modifying gold or silver films on the surface of optical fibers and using their resonance absorption peaks sensitive to the environmental refractive index to achieve the detection of biochemical molecules, such as liquid refractive index [1, 2], biochemical molecules [3, 4], pressure [5], temperature [6], gas concentration [7], humidity [8] and pH [9]. Because of their label-free, fast response and high sensitivity, they are widely used in environmental monitoring, civil engineering, medical diagnosis, food safety and other fields [10]. However, early studies were conducted for the detection of single samples. With the expansion of application fields, the types of samples to be measured have become complex and diverse, and accordingly, the requirements for optical fiber SPR sensors in terms of multi-parameter and accuracy measurements have increased.

In recent years, the measurement of glucose concentration has played an increasingly important role in disease monitoring and science research [11]. Although several glucose concentration detection sensors have become mature, but external temperature can affect the accuracy of glucose concentration measurement [12]. Therefore, its necessary to achieve simultaneous measurement of glucose concentration and temperature without increasing the complexity of the sensing system.

In this paper, we propose a reflective optical fiber probe based on the MSM heterojunction structure. A gold film is sputtered on the surface of the SMF region of the heterojunction, where half of the region is directly used for glucose concentration measurement, and the other region is covered with a PDMS@Au complex for high-sensitivity temperature measurement. A silver film is deposited on the end face of the probe by a silver mirror reaction to collect the reflected light and thus improve the SNR. The proposed reflective optical fiber SPR sensors is not only compact, robust, and cost-effective, but also enable remote detection and real-time monitoring of glucose concentration with high sensitivity and accuracy under temperature-compensated effects.

2. Fiber Probe Fabrication and Sensor Construction

2.1 Fiber Probe Fabrication

The preparation process of the SPR fiber probe is shown in Figure 1. First, two 105/125μm multimode fibers (MMF) and one 9/125μm single-mode fiber (SMF) with a length of 10 mm were cut flat with a fiber cleaver, and their end surfaces were fused with a fiber fusion splicer (AI-8, Zhuoshi, China) to prepare a fiber core mismatch structure. The MMF-SMF-MMF heterojunction fiber could generate evanescent wave in the SMF region for excitation of the SPR effect. Next, the heterojunction fiber was placed in the vacuum chamber of an ion sputterer coater (JS-1600, Saintins, China) and a 40 nm gold film is deposited on the side of the SMF. It is worth noting that in order to achieve uniformity of the coating, it is necessary to flip the fiber over after coating on one side.

In addition, in order to improve the sensitivity, we took a simple silver mirror reaction to deposit a silver film on the end face of the multimode fiber of the fiber probe to achieve light reflection. First, the silver-ammonia solution was prepared by adding 20% ammonia solution drop by drop to 2 mL of 0.1 M silver nitrate ($AgNO_3$) solution, and after the solution changed from brick red to colorless and transparent, 1 mL of 0.8 M sodium hydroxide (NaOH) solution was added drop by drop to create an alkaline environment, and then the ammonia solution continued to be added drop by drop until the solution became colorless and transparent again. The MMF end face was immersed in 0.1% mass concentration of stannous chloride ($SnCl_2$) solution for 5 min to enhance its activity. The prepared silver ammonia solution was dripped onto the MMF end face, and then 0.25 M glucose solution was added slowly to trigger the silver mirror reaction to deposit the silver film, and finally the fiber probe was rinsed repeated with deionized water. This method effectively separates the sensing region and the end face, which can realize the light signal reflection without affecting the thickness of the gold film in the sensing region, and finally improve the detection sensitivity without affecting the detection accuracy.

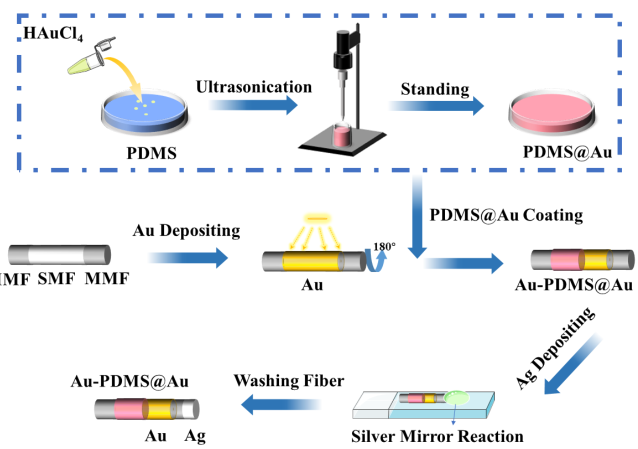

Figure 1. Fabrication scheme of the fiber probe.

Subsequently, we started to prepare temperature-sensitive materials for sensing probes, namely PDMS@Au composites. We used an in-situ reduction method to modify PDMS (Dow Corning). Firstly, PDMS polymer and curing agent were mixed in a 10:1 mass ratio to obtain PDMS precursor solution; next, the prepared PDMS precursor was mixed into 30μL, 100 mM $HAuCl_4$ solution and the configured mixture was subjected to an ultrasonic cell crusher (SCIENTZ-950E, Scientz, China) for Ultrasonic crushing treatment was performed for 10 min. Because of the presence of a large amount of Si-H bonds in the PDMS curing agent, the PDMS curing agent participated in the reaction as a reducing agent to reduce HAuCl4 to Au to achieve uniform distribution of Au in the PDMS colloidal mixture to constitute PDMS@Au composites, which were left to stand for 30 min to remove air bubbles. Finally, the PDMS@Au composite was coated on a gold film coated on a section of heterojunction fiber, and the PDMS@Au composite was heated at 60 °C for 3 h to cure the PDMS@Au composite, completing the preparation of a reflective optical fiber SPR probe for simultaneous measurement of glucose concentration and temperature.

2.2 Sensor Construction

The schematic of the reflective optical fiber SPR sensor is shown in Figure 2. A halogen lamp (HL-2000, Ocean Optics, USA) worked as a broad spectrum light source, and a fiber spectrometer (HR-4000, Ocean Optics, USA) was adopted to analyze the reflected signal light from the fiber probe. Both the halogen lamp and the fiber spectrometer were connected to the fiber probe via a 2×1 fiber coupler with the coupling ratio of 50:50. The reflective sensor structure has a parallel configuration, making it more compact and convenient for remote detection and on-site inspection.

During detection, the heterojunction fiber without gold film and PDMS coating but with silver film coating on the MMF end face was placed in deionized water to record the reference spectrum as I_{ref} with the light source on and the dark spectrum as I_{dark} with the light source off. The fiber probe with gold film and PDMS coating and with silver film coating on the MMF end face were placed in the solution under detection to record the signal spectrum as I_{signal} with the light source on. The SPR spectrum was characterized as $(I_{signal} - I_{dark})/(I_{ref} - I_{dark})$ for refractive index and temperature sensing. All these detecting procedures follow the classical detecting process of SPR sensor.

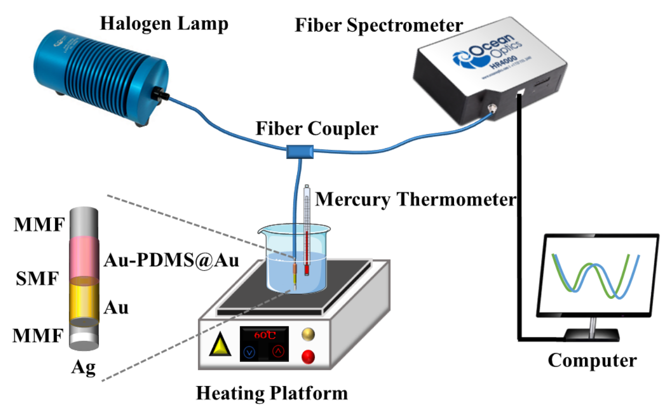

Figure 2. Refractive index measurements with the proposed optical fiber LSPR sensor.

3. Results and Discussion

First, when the glucose concentration C is increased while keeping the temperature T constant, the measurement results are shown in Figure 3(A). There are two resonance dips in the reflection spectra. One is the resonance dip due to the glucose concentration with resonance wavelength of λ_c, and the other due to the temperature with resonance wavelength of λ_T. It is can be seen that the resonance wavelength λ_c undergoes a significant red shift while the resonance wavelength λ_T is unchanged. The unchanged λ_T is mainly due to the isolation of the gold film from the glucose solution by the PDMS@Au complex. Figure 3(B) illustrates the quantitative relationship between them. As seen that the red shift of λ_c is proportional to the change of glucose concentration C with a sensitivity of 3.05 nm/%.

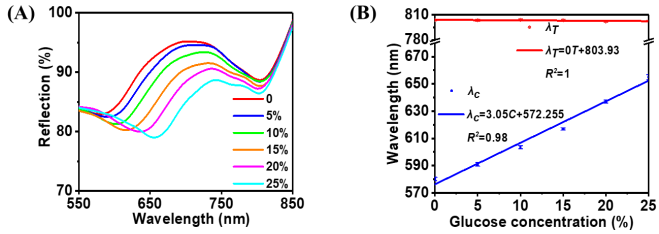

Figure 3. (A) SPR reflection spectra of the dual-parameter fiber sensor immersed in different glucose solutions at a fix temperature of 30°C. (B) Sensitivity fitting curve of the sensor response to glucose concentration.

On the other hand, when the glucose concentration C is held constant and the temperature is increased, the measurement results are shown in Figure 4(A). The resonance wavelength λ_T undergoes a blue shift while the resonance wavelength λ_c only shows a slight change. The small change of λ_c is mainly due to a small change in the refractive index of the glucose solution as a result of the temperature change. The cross-sensitivity may induce error in the measurement of glucose concentration. Figure 4(B) display the quantitative relationships between them. As seen that both of λ_T and λ_c are negatively proportional to the change of temperature with the sensitivities of -1.69 nm /°C and -0.29 nm /°C, respectively.

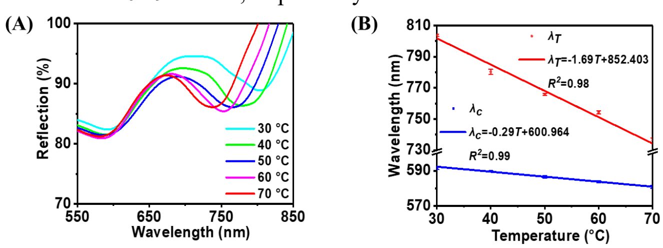

Figure 4. (A) SPR reflection spectra of the dual-parameter fiber sensor immersed in 5% glucose solutions at different temperatures. (B) Sensitivity fitting curve of the sensor response to temperature.

Obviously, the resonance wavelength shift ($\Delta\lambda_c$) is affected by changes in temperature(ΔT) and glucose concentration (ΔC), while the resonance wavelength shift ($\Delta\lambda_T$) is determined only by changes in temperature (ΔT). The corresponding functional relationship can be expressed by the following matrix equation:

$$\begin{bmatrix} \Delta\lambda_c \\ \Delta\lambda_T \end{bmatrix} = \begin{bmatrix} -0.29 & 3.05 \\ -1.69 & 0 \end{bmatrix} \begin{bmatrix} \Delta T \\ \Delta C \end{bmatrix} \quad (1)$$

Finally, the sensing matrix can be expressed as:

$$\begin{bmatrix} \Delta T \\ \Delta C \end{bmatrix} = \begin{bmatrix} 0 & -0.592 \\ 0.328 & -0.056 \end{bmatrix} \begin{bmatrix} \Delta\lambda_c \\ \Delta\lambda_T \end{bmatrix} \quad (2)$$

Therefore, the relationships between the changes of temperature and glucose concentration with the shifts of two resonance wavelengths can be described by:

$$\Delta C = 0.328 \times \Delta\lambda_c - 0.056 \times \Delta\lambda_T \quad (3)$$

$$\Delta T = -0.592 \times \Delta\lambda_T \quad (4)$$

The changes in temperature and glucose concentration were measured by observing the shifts of λ_c and λ_T from the SPR output spectrum in order to solve the cross-sensitivity issue and provide the simultaneous measurement of the temperature and glucose concentration.

4. Conclusions

In this paper, we designed a reflective optical fiber SPR sensor based on MSM heterojunction structure for simultaneously measure the glucose concentration and temperature. The PDMS@Au complex was used to improve the temperature sensitivity. The silver film coated on the end face of the fiber probe was used to improve the sensing SNR. Through theoretical analysis and experimental measurement, the sensitivities for the glucose concentration and temperature were demonstrated to be 3.05 nm/% and -1.69 nm/°C, respectively. The proposed reflective optical fiber SPR sensors is not only compact, robust, cost-effective, and easy to fabricate and manipulate, but also enable remote detection and real-time monitoring of glucose concentration or other analytes with high-sensitivity and accuracy under temperature-compensated effect. It has favorable application potential in the fields of food safety, biochemistry, and environmental monitoring.

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