

## Regular Articles

## Graphene enhanced optical fiber SPR sensor for liquid concentration measurement

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## ABSTRACT

A high sensitivity optical fiber Surface Plasmon Resonance (SPR) sensor which based on coreless optical fiber, silver film and graphene, has been designed and implemented for liquid concentration detection. In this paper, Graphene is firstly verified that it can be used to enhance the evanescent field of traditional optical fiber and thus increasing sensitivity in experiment. The sensitivity of proposed sensor is 6.417 nm/%, which is higher than that of the traditional optical fiber SPR sensor according to the comparative experiments. In addition, the proposed sensor is extremely easy to make and the silver film could be protected from oxidation and damage due to the existence of graphene. Moreover, the sensor has pretty small size, immunity to electromagnetic interference, quick response speed and thus can suitable a variety of severe environments and real-time measurement.

## 1. Introduction

Graphene has received widespread attention in recent years due to its adsorption capacity, bio-compatibility, nanometer level size, and immunity to electromagnetic interference. And it has been applied to many fields, such as transistors, solar cells, display panels, capacitors and other electronic devices [1]. Besides, the optical properties of universal broad band optical, plasmonic properties and high RI [2,3] have also made graphene useful in optics. And nanoscale structure with extremely high surface-to-volume ratio makes graphene be integrated into an optical fiber sensor in many ways, for example, as a thin sensitive film deposited onto the fiber [4] and as a photo-detecting material [5]. Moreover, a photonic operating mechanism has a faster response, so graphene employed to optical sensor can realize faster response time and higher sensitivity [6].

The optical fiber SPR sensors have many advantages, such as high sensitivity, label free, non-destructive and real-time detection, and so on. Thus, they are widely used in the studies of biomolecular interaction analysis and RI measurement, even health-care, environment monitoring, food safety and homeland security [7–12]. The optical sensors that combine graphene with the SPR effect have been proposed [13,14,15], even it has been confirmed that graphene coating fiber SPR sensor can improve the sensitivity of the sensor in theory [15]. But experimental verification is not sufficient and the existing sensors, such as using fiber Bragg-gratings, long period fiber gratings, tilted fiber Bragg gratings, Fabry–Perot interferometers, Mach–Zehnder interferometers, micro-fiber Sagnac interferometers and so on, have some

disadvantages more or less, such as low sensitivity and complicated device fabrication processes, poor mechanical strength, etc.

In this paper, concentration measurement system is designed with end reflection optical fiber SPR sensor. One end of the coreless optical fiber is coated by Ag film to form a simple SPR sensor. And graphene is deposited on the outside of the proposed optical fiber SPR sensor for a comparative experiment. It is the first time to verify that graphene can improve the performance of sensor experimentally. The proposed optical fiber SPR sensor can be easily used in measuring refractive index or liquid concentration with many advantages, such as extremely simple, easy to measure, high sensitivity, small size, long distance detection, and potential application to other fields.

## 2. Principle of sensing

The proposed end reflection optical fiber SPR sensor with graphene is shown in Fig. 1. The sensor is composed of the coreless fiber with diameter  $D$ , Ag film with thickness  $d_1$ , and graphene film with thickness  $d_2$ . And around the graphene film is the sensing medium to be measured. The light from a collimated source is launched into one end of the fiber at the axial point. Total internal reflection (TIR) will occurred in the optical fiber, which means that prism coupling principle of SPR is still valid in the optical fiber.

And the light transmitted in the fiber based on TIR, whose total reflection angle is  $\theta$ . Arriving at the end of the fiber, the reflecting mirror, the light will return to the incoming end. The power,  $dP$ , arriving at another end of the fiber between the incident angles  $\theta$  and

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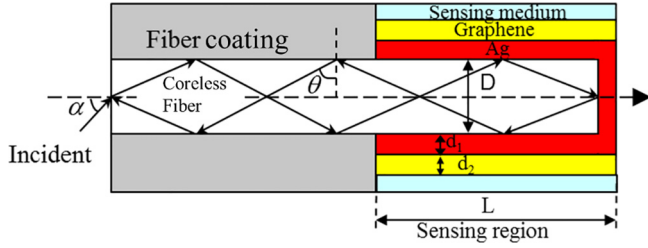


Fig. 1. Schematic diagram of the proposed end reflection optical fiber SPR sensor with graphene.

$\theta + d\theta$  could be written as [16]

$$dP \propto P(\theta)d\theta \quad (1)$$

where the mode power corresponding to the incident angle  $\theta$  is

$$P(\theta) = \frac{n_1^2 \sin^2 \theta \cos \theta}{(1 - n_1^2 \cos^2 \theta)^2} \quad (2)$$

where  $n_1$  is the RI of the coreless fiber. If the resonance of fiber end face is ignored, the normalized reflected power of p-polarized light will be derived as Eq. (3), by using the reflectance value for a single reflection at the coreless fiber/metal interface.

$$R_{ref} = \frac{\int_{\theta}^{\pi/2} R_p^{N_{ref}(\theta)} P(\theta) d\theta}{\int_{\theta}^{\pi/2} P(\theta) d\theta} \quad (3)$$

where  $N_{ref}(\theta) = 2L/D \tan \theta$  is the total number of light reflected in the sensing region, and  $L$  is the length of sensing region,  $D$  is the diameter of the coreless fiber.

The reflection intensity for p-polarized light is expressed as [17]:

$$R_p = |r_p|^2 \quad (4)$$

The amplitude reflection coefficient for p-polarized incident wave is given by,

$$r_p = \frac{(M_{11} + M_{12}q_N)q_1 - (M_{21} + M_{22}q_N)}{(M_{11} + M_{12}q_N)q_1 + (M_{21} + M_{22}q_N)} \quad (5)$$

where  $M = \prod_{k=2}^{N-1} M_k$ ,  $M_k = \begin{bmatrix} \cos \beta_k & -i \sin \beta_k / q_k \\ -i q_k \sin \beta_k & \cos \beta_k \end{bmatrix}$ ,  $q_k = \frac{(\epsilon_k - n_1^2 \sin^2 \theta)^{1/2}}{\epsilon_k}$  and

$\beta_k = \frac{2\pi d_k}{\lambda} (\epsilon_k - n_1^2 \sin^2 \theta)^{1/2}$ .  $N$  is the layers of the sensor. The proposed end reflection optical fiber SPR sensor has to be analyzed using the four layers (coreless fiber/metal/sensing layer/sample) model. In addition, the RI and dielectric constant can be written as [18]:

$$\epsilon = n^2 \quad (6)$$

In the present study simulations, the complex RI of graphene is given by [19]:

$$n_3 = 3 + i \frac{c}{3} \lambda \quad (7)$$

where  $\lambda$  is the wavelength in  $\mu\text{m}$  and the constant  $c$  is equal to  $5.446 \mu\text{m}^{-1}$ . The thickness ( $d_2$ ) of single layer graphene is 0.34 nm.

The performance of the proposed end reflection SPR fiber sensor can be simulated by the above theory. As shown in Fig. 2 and Fig. 3, the mode field of SPR has been calculated by COMSOL 5.2.

From Figs. 2 and 3, the optical fiber SPR sensor with graphene has bigger effective RI than traditional optical fiber SPR sensor. And the mode field is closer to sensing medium. So, it can be considered that the sensor with graphene has a stronger evanescent field and thus this is more sensitive to measure surrounding refractive index (SRI).

According to the structure diagram of sensor, the reflective spectra of the proposed end reflection optical fiber SPR sensor with different SRI. The diameter of coreless fiber is 600  $\mu\text{m}$ , the thickness of Ag film is 40 nm, and monolayer graphene is used in the sensor with 0.34 nm.

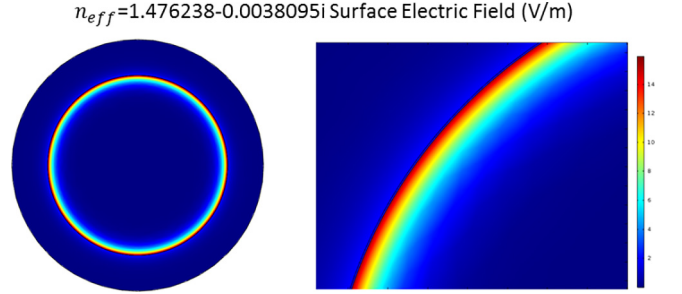


Fig. 2. The mode field of traditional optical fiber SPR sensor.

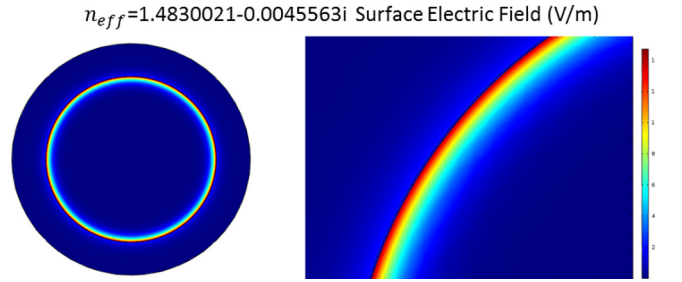


Fig. 3. The mode field of optical fiber SPR sensor with graphene.

And the SRI ranges from 1.3411 to 1.3737. Fig. 4 shows the reflectivity spectra of the proposed end reflection optical fiber SPR sensor with varied SRI, and the relationship between the resonant wavelength and SRI are shown in the inset. It can be seen that the resonance wavelength is red-shifted with increasing SRI. Comparing with Fig. 4(a) and (b), the sensitivity of traditional SPR sensor reaches 2657 nm/RIU, but the sensitivity of the proposed sensor with graphene gets 3091 nm/RIU. It means that graphene can improve the sensitivity of SPR sensor effectively. The reason is that the evanescent field has been strengthened between graphene film and sensing medium due to the high RI of graphene. And the maximum measured value of SRI can reach 1.45, equal to the RI of the coreless fiber.

### 3. Experiments and results

The concentration measurement system is schematically shown in Fig. 5. Plastic clad silica optical fiber with 600  $\mu\text{m}$  diameter and 0.37 numerical aperture purchased from Beijing Glass Research Institute is adopted in this paper. The sensing part (about 10 mm) of the probe is dipped in the acetone to soften the cladding layer, and then the cladding layer of the sensing part is removed easily. And Ag film coats the sensing part of coreless fiber by the chemical method proposed in [20]. The basic principle of this chemical method is the Tollens reaction between silver ammonia solution and glucose solution. Firstly, silver ammonia solution was freshly prepared. 2 mL  $\text{AgNO}_3$  (0.1 mol/L) was put into a beaker and mixed by a magnetic mixer and the ammonia (20%) dropped until the solution was clarified. And then, the ammonia (20%) was dropped into the beaker after adding 1.4 mL KOH (0.8 mol/L) until the solution is clarified again. By now, the silver ammonia solution was obtained. Secondly, 0.4 mL glucose solution (0.25 mol/L) was added to the silver ammonia solution with slight stirring, then the coreless fiber was put into the solution quickly. Finally, the surface of fiber and the inner wall of the beaker formed a layer of shiny silver after resting for a while. As we all know that the generated silver is not suspend in the solution but attached to the object surface in Tollens reaction. The concentration of reaction solution and reaction time were determined by many experiments. After coating the cylindrical surface of the sensing part with Ag film, the traditional SPR sensing probe was completed. For contrast experiment, two identical sensing probes were made at the same time, and then one of the probes were deposited with

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