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Three-layer-structure polymer optical fiber with a rough inter-layer surface as a highly sensitive evanescent wave sensor

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ABSTRACT

We present a high-sensitivity and high-accuracy D-shaped polymer fiber-optic evanescent wave (FOEW) sensor with a three-layer structure (i.e., bottom, inter-, and surface layers) and a rough inter-layer surface in the sensing region. The surface layer is made of dilute Canada balsam in xylene, and the longitudinal section is coated with light-absorbing (LA) film. To obtain the maximum sensitivity and accuracy, the morphologies of the inter-layer and surface layer are determined. The effects of coating thickness and the refractive index of the surface layer and LA film on the performance of FOEW sensors are investigated both theoretically and experimentally. The sensitivity, accuracy and repeatability of the sensors are evaluated using glucose solutions; the response of the sensors, which are self-assembled from the polycation (tris[2-(4-phenyldiazenyl) phenylaminoethoxy] cyclotriveratrylene (TPC) plus poly dimethyl diallyl ammonium chloride (PDDAC)) and polyanion (TPC plus polyacrylic acid (PAA)), to Hg(II) in aqueous solutions is examined. The results highlighted the high sensitivity of the FOEW sensor with a three-layer structure and appropriate roughness of the inter-layer surface, which showed a 6.5-fold improvement in the detection of the target Hg(II) ions compared to the conventional FOEW sensor with a core-cladding structure, and the novel sensor was validated with a lower limit of detection of 0.1 mg L^{-1}.

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1. Introduction

Fiber-optic evanescent wave (FOEW) sensors are based on the interaction of an analyte with an evanescent field of light through an optical fiber, and their measurements are not affected by the bulk solution because the penetration depth of the evanescent field ranges from ten to several hundred nanometers [1–8]. Furthermore, FOEW sensors are more sensitive than attenuated total reflection (ATR) point sensors because of the multiple reflections that occur within a short sensing region [9,10]. Thus, FOEW sensors, which are composed of polymer and silica optical fibers, have been widely applied in chemistry, biochemistry, life sciences, and environmental research [11–19]. Although FOEW sensors present numerous advantages and represent a promising technology, they still have two critical flaws: low sensitivity and accuracy.

Various techniques to enhance the sensitivity of FOEW sensors have been developed. One of the simplest and best-known techniques is based on the optimization of the shape of the unclad fiber region and the fiber end region to increase the evanescent field decay [18–23]. Although the sensitivity is improved by optimizing the shapes of the unclad fiber sensing region and unclad fiber end region, the unclad fiber surface exhibits a certain degree of roughness induced by the grinding and etching used during fabrication. Interestingly, Zhong et al. [23,24] and Zhuang et al. [25] have discovered that although surface defects in the sensing region of the fiber increase the light-scattering loss, thus reducing the effectiveness of optical signal transmission, sensor surfaces with an appropriate roughness exhibit higher sensitivity. Although optimized shapes and surface roughness can further improve the sensitivity of FOEW sensors, the number of points at which ATR occurs does not lend itself to further improvement using the standard (traditional) fibers with normal core-clad structures. Thus, based on the currently designed FOEW sensors, to further enhance the sensitivity and specificity, surface plasmon resonance (SPR) and fluorescence technologies have been coupled to FOEW sensors. In particular, fluorescence technology for detection of metal ions has been introduced by multiple researchers [2,4,8,12,15–17,19,22,26–35]. Although opti-

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mized shapes plus metal ion sensitive films can further improve the sensitivity and selectivity of FOEW sensors [2,8,15–17,19,22,36,37], fiber-optic fluorescence sensors still depend on the evanescent field intensity at the surface of the unclad fiber. Thus, enhanced initial evanescent field intensity is very important for developing high-sensitivity FOEW sensors.

It is well known that fibers with core-clad structures were developed for the transmission of information in telecommunication, and core-clad structures with core-clad waveguides are used to maintain the standard optical transmission modes and reduce the interaction between the light and the environment [38]. Thus, the conventional core-clad-structure FOEW sensors show very low evanescent field intensity because of the limited number of points at which ATR occurs. Hence, it is difficult to further enhance the evanescent field intensity of FOEW sensors using traditional fibers. Furthermore, for conventional FOEW sensors, especially D-shaped FOEW sensors, the measurements of the sensors are controlled by the light rays from the surfaces of the cross-section (C_I in Fig. 1) and the sensing region (Fig. 2). The incident light at the surface of the sensing region will be diminished by evanescent field absorption and by the decay of the higher-order modes [39–41]; the reduction of light intensity increases with the refractive index (RI) or with the concentration of analyte, which decreases the output light intensity and increases the sensitivity of FOEW sensors. However, when the incident light is transmitted to the surface of the cross-section, the light passes through the analyte and will be coupled into the fiber at the surface of the sensing region. This increases the output light intensity of the FOEW sensors, interferes with the measurement of light attenuation, and degrades the accuracy and sensitivity of the sensor. Unfortunately, the conflicting results have not been resolved. Thus, it can be concluded that if standard fibers continue to be applied in the fabrication of D-shaped FOEW sensors, it will be difficult to overcome the fundamental problem of low sensitivity and accuracy. Development of a novel fiber structure could provide a powerful means to resolve the problem of the sensitivity and accuracy of the D-shaped FOEW sensors.

In this work, a novel D-shaped polymer FOEW sensor with a three-layer structure and a rough inter-layer surface for detection of Hg(II) ions has been fabricated. A schematic of the preparation process and mechanism is shown in Fig. 1. To increase the number of ATR points, which increases the sensitivity of the sensors, the standard polymer optical fiber (POF) is replaced by a three-layer structure and a rough inter-layer surface in the sensing region. The novel sensing region of the proposed sensor is composed of bottom, inter-, and surface layers as shown in Fig. 2. The refractive index (RI) of the bottom layer, $n_1 = 1.406$, is less than that of the fiber inter-layer, $n_2 = 1.492$ (the bottom layer and second-layer are POFs composed of standard cladding, i.e., fluorinated polymer and a fiber with a poly(methyl methacrylate) (PMMA) core, respectively). Because the surface-layer is made of dilute Canada balsam in xylene (CBX), its RI, $n_3 = 1.524$, is greater than n_2. To eliminate the background noise and enhance the accuracy of the sensor, the cross-section that is closest to the light source is coated with light-absorbing (LA) film composed of black paint doped with carbon nanotubes. Furthermore, to enhance the sensitivity and specificity, Hg(II)-sensitive film was prepared by employing a layer-by-layer self-assembly approach and using tris[2-(4-phenyldiazenyl) phenylaminoethoxy] cyclotriveratrylene (TPC) chromophore, poly dimethyl diallyl ammonium chloride (PDDAC) polycation and polyacrylic acid (PAA) polyanion. To obtain the maximum sensitivity and accuracy, we examine the inter-layer, surface layer and Hg(II)-sensitive film morphologies and perform an experimental investigation on the effects of inter-layer surface roughness, LA film, and surface layer thickness on the light transmission and sensitivity of these POF sensors. We also perform a theoretical investigation of the effects of surface layer thickness and RI on the performance of POF sensors. In addition, the performance of the FOEW sensors created for this study is evaluated using glucose and Hg(II) ion analytes.

2. Light transmission and sensitivity analysis of the sensor

In this work, to enhance the sensitivity of the FOEW sensors, different POFs with rough inter-layer surfaces in the sensing region have been prepared by employing different diamond lapping films. The effects of surface roughness on the optical properties and sensitivity of fiber-optic sensors have been previously reported [23,24]. A summary of the previously published work can be stated as follows: The transmitted light intensity of the thinned fibers decreases with increasing surface roughness, whereas the intensity of the evanescent waves (EWs), the EW decay coefficient, EW penetration depth, and EW optical path length on the unclad fiber surface increase with increasing surface roughness. Hence, the sensitivity of the fiber-optic sensors initially increases and then decreases with increasing roughness; in particular, for a graded-index multimode silica optical fiber, the highest sensitivity can be obtained at an appropriate roughness. Although the effects of roughness on the performance of conventional core-clad structure FOEW sensors have previously been studied [23,24], the effects of the cross-section and surface-layer parameters on the optical properties and sensitivity of polymer FOEW sensors with a three-layer structure have not been investigated. Thus, to clarify the effects of the cross-section and surface-layer parameters in the sensing region on the performance of the D-shaped three-layer-structure FOEW sensors, we have performed a theoretical investigation of the effects of these parameters on the light transmission and sensitivity of the sensors. A detailed theoretical analysis has been presented in Appendix A of the Supplementary material, and the representative ray paths are presented in Figs. 2a–b and 1s of the Supplementary material. The simulation parameters and results are listed in Table 1.

In Table 1, $U_{i,j}$ ($j = 1–4$) is the angle of light rays on the input end of the fiber; l is the free-space wavelength of the light; r_1 is the diameter of the sensing region of the conventional D-shaped POFs; r_2 is the diameter of the thinned D-shaped POFs with the three-layer structure; k is the surface layer thickness; L is the length of the sensing region of the sensors; n_0 is the RI of air; n_2 is the RI of the fiber core; n_3 is the RI of the surface layer; n is the RI of the analyte; N is the number of reflections of light rays (I_3) in the sensing region (Fig. 2a); N' *is* the number of reflections of light rays (I_4) in the sensing region (Fig. 2b); N'' is the number of reflections of light rays (I_2) in the surface layer (Fig. 2b); Dq_c and $Dq'\ c$ represent a small change in the total reflection critical angle for a conventional D-shaped POF and a D-shaped POF with the three-layer structure, respectively; DD_P and $DD'\ P$ represent a small change in the penetration depth of the evanescent wave for a conventional D-shaped POF and a D-shaped POF with the three-layer structure, respectively; DV and DV' represent a small change in the V-parameter for a conventional D-shaped POF and a D-shaped POF with the three-layer structure, respectively; Dq_3 and Db_3 represent a small change in the angle of the coupled light beams for a conventional D-shaped POF and a D-shaped POF with the three-layer structure, respectively.

Table 1 demonstrates two important conflicts: (1) One can see that N' and N'' of the three-layer-structure FOEW sensors show improvement when k is 50–250 mm in comparison to conventional core-cladding-structure sensors when the thinned diameter (r_1) is 650 mm. Furthermore, compared with conventional FOEW sensors, the increment of the critical angle in the three-layer-structure POFs is smaller, i.e., $Dq'\ c < Dq_c$. As the increment of the critical angle decreases, more near-cutoff modes will be attenuated in the sensing region; hence, for the three-layer structure POFs, more high-order or near-cutoff modes can be used to detect analyte at the

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