

Fiber-optic surface plasmon resonance-based sensor with AZO/Au bilayered sensing layer

Woo-Hu Tsai (蔡五湖)², Kuen-Cherng Lin (林坤成)^{1*}, Shu-Ming Yang (杨肃铭)²,
Yu-Chia Tsao (曹育嘉)², and Ping-Jing Ho (何秉琛)¹

¹Department of Electrical Engineering, China University of Science and Technology, Taipei, China

²Graduate Institute of Electro-Optical Engineering, Tatung University, Taipei, China

*Corresponding author: kclin@cc.cust.edu.tw

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We describe a surface plasmon resonance-based fiber sensor based on a side-polished graded-index multimode fiber, in which an Al-doped zinc oxide/gold (AZO/Au) bilayer is deposited on the side-polished surface of the fiber core to improve the detection sensitivity of the device. The AZO/Au layer is used as the active sensing member of the device with a combination of a 75-nm-thick AZO layer and a 40-nm-thick Au layer. Such a device is then applied to the concentration measurement of CH₃COONa solutions, as an example showing a good response to concentration variation. The results indicate that the additional AZO layer in the active sensing member may lead to higher detection sensitivity and greater measurement stability in the measurements of solution concentration.

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Recently, surface plasmon resonance (SPR) phenomena have been the focus of considerable attention in optical sensor applications because of their high sensitivity, flexibility of real-time measurement, and label-free chemical and biological sensing^[1–3]. In addition, optical fibers have been applied to develop various SPR-based sensors because of their advantages such as electromagnetic immunity, compactness and lightness, remote sensing capacity, and durability in adverse circumstances^[3–6]. Of which, multimode fibers, with a much larger core diameter than single-mode fibers, are polished along their axes, so that part of the core and cladding are removed from their cylinder side to expose a proper area of the core. The side-polished core area is then coated with a metal layer, such as a gold (Au) film, to form a SPR sensing element thereon, once the metal layer is surrounded by a dielectric sensing medium^[7–13]. Such side-polished SPR fiber sensors are based on graded-index multimode (GIMM) fibers, which can alleviate fabrication complexity in side polishing, as compared with the ones based on single-mode fibers. Although the SPR fiber sensors can also be structured on step-index multimode fibers, their SPR response performance cannot compete with the ones based on GIMM fibers^[7–9]. The side-polished fiber sensor provides a simple scheme for chemical and biological sensing with high sensitivity in wavelength interrogation^[11] as well as high detection limit for biomolecules^[12]. In addition, the side-polished fiber sensor has been applied in the fields of time-dependent measurements in environmental monitoring^[9] and nanoscale film deposition^[13].

Al-doped or Ga-doped metal oxides, such as Al-doped zinc oxide (AZO) and Ga-doped zinc oxide, have been suggested as indium-free and low-cost electrode materials for solar cell devices^[14], in which an oxide/metal multilayer structure has been proposed as a transparent electrode. In addition, an indium tin oxide coating has been used to support lossy mode resonances

(LMRs) in the LMR-based fiber sensor to mitigate complex surface interferences because of electronic interband transitions^[15]. Other materials like TiO₂ have been tested on side-polished optical fibers for refractometer applications^[16]. We assumed that an outer oxide layer over the metal layer on the side surface of the side-polished SPR fiber sensor may improve its sensitivity performance. In a conventional side-polished SPR fiber sensor, only a single metal layer is applied on the polished side surface to construct the sensing element. In this letter, we describe a fiber-optic SPR sensor with an additional AZO layer overlaying the sensing element of the conventional one. To our knowledge, this is the first time that an AZO-coated side-polished SPR fiber sensor has been applied and characterized to upgrade sensitivity performance. Moreover, the SPR fiber sensor will be applied to concentration measurement of sodium acetate (CH₃COONa) solutions, as an example showing a good response to concentration variation.

The proposed SPR fiber sensor is composed of a side-polished multimode fiber with AZO and Au layers deposited on the polished cylinder side of the fiber, as shown in Fig. 1. The fiber is a GIMM fiber with a core size of 62.5 μm and a cladding diameter of 125 μm , commercially available from the Prime Optical Fiber Corporation. A portion of the cladding and core of the fiber is side-polished to have a flat side surface, on which the Au and AZO layers are to be deposited in sequence. A V-groove channel is formed on a silicon substrate as a holder of the bared fiber to be side-polished. The silicon substrate is a chip with a length of 5 mm divided from a silicon wafer, and the V-groove has a width of 125 μm . The bared fiber is bonded onto the V-groove and the silicon substrate together with the GIMM fiber is then polished by using polishing diamond films of 6, 1, and 0.1 μm grain sizes in sequence^[8]. The side surface is set to have a width of 125 μm and a depth of 62.5 μm down to the fundamental mode region in the fiber core, so that

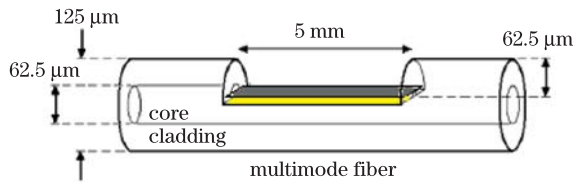


Fig. 1. Proposed SPR-based side-polished fiber sensor.

the surface plasma waves can be excited mainly in the fundamental mode of the multimode fiber.

An Au layer is first deposited on the side surface of the fiber core and an AZO layer is then deposited on the Au layer. The Au layer is formed by using the direct current (DC) sputtering system (ULVAC, Japan) with a chamber pressure of 20×10^{-2} Torr as well as a DC power of 5 V and 20 mA. The AZO layer is formed by using the radio frequency magnetron sputtering system (CESAR 136) with a chamber pressure of 20×10^{-2} Torr and a gas discharge of 10 sccm. As compared with the control group side-polished SPR fiber sensor with only one single sensing layer of Au^[7], the additional AZO layer in this study is used to increase the sensitivity of the SPR-based sensors for refractive index variation of a testing solution. The suggested Au thickness for the SPR effect is approximately 40 nm in our previous works^[10,11], and experiments on the effects of different AZO thicknesses on SPR performance are still under way. In the present letter, a bilayered sensing structure with a 75-nm-thick AZO layer over a 40-nm-thick Au layer is used to explore the key characteristics of the proposed SPR fiber sensor.

Considering an output transmittance spectrum obtained from a light beam launched into a SPR-based sensor with a sensing layer of refractive index n_s , a sharp dip can be observed at its resonance wavelength λ_{res} because of efficient energy transfer from the light beam to the surface plasma. A change in refractive index of the sensing layer from n_s to $n_s + \delta n_s$ may induce a shift in resonance wavelength λ_{res} by $\delta \lambda_{res}$. Thus, the detection sensitivity can be defined as $S = \delta \lambda_{res} / \delta n_s$ ^[17], which means that the sensitivity of a SPR-based sensor depends on the shift of the resonance wavelength responding to the change in the refractive index of the sensing layer. In the experiments, the optical characteristics of the fabricated SPR fiber sensor were measured by using a halogen white light source (Ando AQ4303B) and an optical spectrum analyzer (Ando AQ6315A).

To explore the device response to refractive index variation, the proposed SPR fiber sensor, with a 75-nm-thick AZO film over a 40-nm-thick Au film to form a bilayered sensing layer, was used to measure CH_3COONa solutions of different concentrations. Absorbing solutions like CH_3COONa have a significant imaginary part of the refractive index. The CH_3COONa solutions lack a measurable variation in refractive indices. Hence, the refractive index variation is represented by the concentration variations in the experiment. Figure 2 shows the transmittance spectrum of the proposed SPR-based fiber sensor in the measurements of CH_3COONa solutions with different concentrations. The SPR dip wavelength of the present fiber sensor shifts from 1097.1 nm for the 0-mol/L CH_3COONa solution to 1146.5 nm for the 2.5-mol/L CH_3COONa solution. In addition, a side-polished SPR fiber sensor of the control group with only one sin-

gle sensing layer of Au was fabricated and measured for comparison. Figure 3 shows the transmittance spectrum of the control group SPR-based fiber sensor in the concentration measurements of CH_3COONa solutions. The SPR dip wavelength of the control group sensor shifts from 628.5 nm for the 0-mol/L CH_3COONa solution to 667.2 nm for the 2.5-mol/L CH_3COONa solution.

The relationships of the SPR dip wavelength to the CH_3COONa solution concentration are shown in Fig. 4 for the case of the proposed SPR fiber sensor having a 40-nm-thick Au/75-nm-thick AZO sensing layer and in Fig. 5 for the control group sensor having only a 40-nm-thick Au sensing layer. We observed a linearly proportional relationship between the SPR dip wavelength and the solution concentration. Slopes of the linear fitting lines represent the sensitivity of the sensors to the concentration of the CH_3COONa solution. The sensitivity of the proposed SPR fiber sensor versus the CH_3COONa solution concentration is 20.3 nm/(mol/L), larger than that (15.3 nm/(mol/L)) of the control group sensor. As shown in Figs. 4 and 5, the proposed Au/AZO-coated device and the control group Au-coated device have their resonances in different regions of the spectra. Although the sensitivity variations attributed to different sensitivities of the spectrometer in the different regions may be minor, it still suggests that the added AZO layer in the presently proposed device would be capable of improving the detection sensitivity of the side-polished SPR-based fiber sensor for the measurement of solution concentration. In addition, the detection accuracy of a SPR-based sensor depends on how precisely it can detect the dip wavelength of resonance. The detection accuracy can be defined as a signal-to-noise ratio (SNR) = $\delta \lambda_{res} / \delta \lambda_{1/2}$,

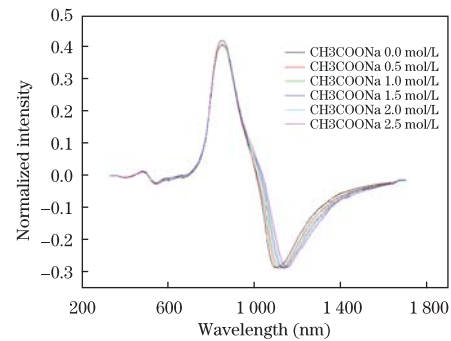


Fig. 2. Transmittance spectrum of the proposed SPR-based fiber sensor in the measurements of CH_3COONa solutions with different concentrations.

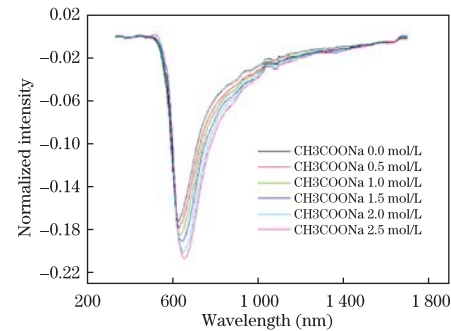


Fig. 3. Transmittance spectrum of the control group SPR-based fiber sensor in the measurements of CH_3COONa solutions with different concentrations.

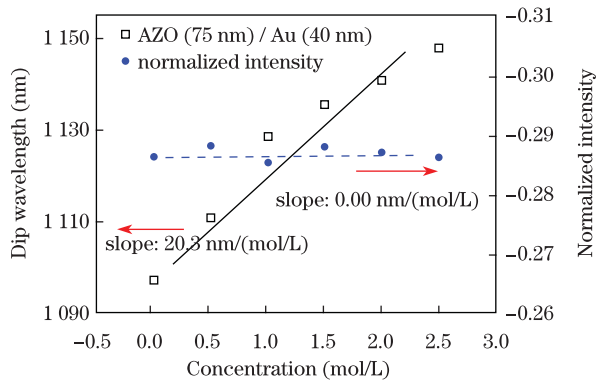


Fig. 4. Relationship of the normalized intensity at the SPR dip wavelength to the CH_3COONa solution concentration for the case of the proposed SPR-based fiber sensor.

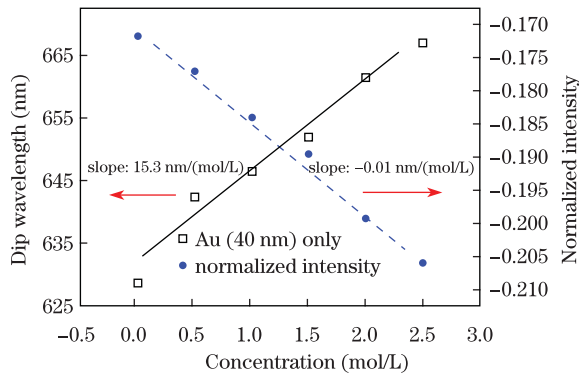


Fig. 5. Relationship of the normalized intensity at the SPR dip wavelength to the CH_3COONa solution concentration for the case of the control group SPR-based fiber sensor.

which is inversely proportional to $\delta\lambda_{1/2}$, the spectral width of the transmittance spectrum corresponding to a reflectance of one half^[6]. The SNR can be evaluated to be approximately 0.3 based on Fig. 2.

The detection accuracy of a SPR-based sensor depends on how precisely it can detect the resonance wavelength and, hence, the refractive index of the sensing layer. The narrower the dip width of the transmittance spectrum, the higher the detection accuracy. Thus, the detection accuracy can be defined as the $\delta\lambda_{\text{res}}/\delta\lambda_{1/2}$, which is inversely proportional to $\delta\lambda_{1/2}$, the spectral width of the transmittance spectrum corresponding to a reflectance of one half. Moreover, the normalized intensity at the SPR dip wavelength of the presently proposed fiber sensor keeps almost constant for the CH_3COONa solution in the concentration range from 0 to 2.5 mol/L, while the normalized intensity at the SPR dip wavelength of the control group sensor decreases from -0.172 for the 0-mol/L CH_3COONa solution to -0.206 for the 2.5-mol/L CH_3COONa solution. This finding implies that the addition of the AZO layer may help lower the measured intensity variation at the SPR dip wavelength of the SPR-based fiber sensor, which is conducive to good measurement stability. Thus, the proposed SPR fiber sensor shows better detection sensitivity and measurement stability, as compared with the control group sensor without the AZO layer.

In conclusion, a SPR-based fiber sensor based on a side-polished GIMM fiber is described. An AZO layer is formed on the Au film of the conventional device, so

that the AZO/Au bilayer is used as the active sensing member to improve the device's detection performance. The proposed sensor is then applied to the concentration measurement of CH_3COONa solutions, as an example showing a good response to concentration variation. A sensitivity of 20.3 nm/(mol/L) is obtained in the proposed device, which is better than 15.3 nm/(mol/L) of the conventional device of the control group. The normalized intensity at the SPR dip wavelength of the proposed device keeps almost constant for the solution concentration range from 0 to 2.5 mol/L. The results indicate that the added AZO layer in the active sensing member may lead to higher detection sensitivity and greater measurement stability in the measurements of solution concentration. These findings would provide side-polished SPR-based fiber sensors with potential applications in the chemical and biological sensing fields. Furthermore, considering that the SPR and LMR phenomena may originate from the AZO layer^[18], additional experiments are scheduled to clarify the attribution of the device's performance enhancement and will be reported in our future publications.

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