A compact, flexible fiber-optic Surface Plasmon Resonance sensor with changeable sensor chips

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10.1016/j.snb.2017.02.064
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A R T I C L E  I N F O

Article history:
Received 10 November 2016
Received in revised form 6 February 2017
Accepted 10 February 2017
Available online 16 February 2017

Keywords:
Fiber optic sensor
Surface Plasmon Resonance

A B S T R A C T

We propose and demonstrate the concept of a novel compact, flexible fiber optic Surface Plasmon Resonance (SPR) sensor based on a double-pass Kretschmann-type configuration, where the SPR sensor chip can be replaced for various sensing applications. Simulation and experimental results demonstrate that the proposed fiber-optic SPR structure has a sensitivity to salt concentration of around 4.8 μW/ppt.

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1. Introduction

Surface Plasmon Resonance (SPR) is a high-sensitivity optical sensing technique that is used for the real-time detection of small changes in the effective refractive index of metal-dielectric interfaces. SPR has been widely used for real-time monitoring of bio-molecular interactions and detecting chemical and biological analytes in liquid or gas media. This technique is based on the interaction between light and free electrons of the semitransparent noble metal layer (or chip). At resonance conditions, the frequency of incident light matches the oscillation frequency of the metal’s electrons, hence the intensity of the light reflected off the metal layer decreases significantly, and a surface plasmon wave is generated through photon energy transfer into the thin metallic layer. Silver and gold are the preferred metals for triggering SPR due to the fact that they are chemically stable while providing reasonable sensitivity to refractive index changes [1–5].

Fiber optic sensors have become attractive sensing candidates due to several unique features, such as immunity to electromagnetic interference, high sensitivity, and small foot print, which enables them to be integrated with a multiple-fiber sensing platform for the simultaneous detection of many compounds.

With the increasing demand for in-situ and real-time monitoring for environmental, industry process, gas, food, biomedicine, and health applications, the miniaturization of SPR sensors has recently attracted great attention. Several fiber optic techniques have been reported for the realization of compact SPR sensor devices [6–8]. These fiber optic SPR sensors involve complex fabrication processes such as chemical etching and evaporation of metallic materials along the fiber core. Usually the metallic sensing layer has a short life span that depends on the environment within which the sensor operates. In harsh environments, the performance of the fiber optic SPR sensor dramatically decreases when the quality of the metallic sensing layer degrades, necessitating the disposal of the whole sensor.

Additionally, sensors based on the use of planar waveguides have been reported [9–11]. This type of sensors are typically used for biosensing since they enable the detection of different types of analytes with high resolution. In particular, sensors based on the use of metal-clad waveguides (MCWGs) have (i) narrower full-width half maximum (FWHM) shape dips than their SPR sensor counterparts, making them attractive for analysing analytes [12], and (ii) longer lifetime, due to the use of a chemical barrier that protects the metal nano-layer [13].

In this paper, we propose and demonstrate a novel compact, flexible fiber optic sensor featuring simple fabrication with changeable SPR sensor chips. Experimental results show that the proposed fiber optic SPR sensor is able to detect changes in the refractive index of water samples of salinities with a sensitivity of 4.8 μW/ppt. To the best of our knowledge, the proposed fiber optic SPR sensor is the first compact sensor that offers SPR sensor chip replacement, and this opens the way for the development of multi-functional SPR sensor platforms.

2. Proposed fiber-optic SPR sensor structure

Fig. 1 shows the proposed fiber optic SPR sensor structure, which comprises a fiber optic polarisation maintaining collimator with the
following specifications of a 1.8 mm in diameter size C-type lens, a working distance of 10 cm, a beam diameter of ~0.4 mm and a working wavelength range of 1300–1900 nm. Also, micro-prism of cross section dimensions 5 mm × 5 mm × 4.46 mm and height 5 mm was made using BK7 glass. A high reflectivity 100 nm silver layer was deposited onto one of the micro-prism’s sides using an e-beam evaporation system. Furthermore, a re-attachable silver nano-thin-film SPR sensing chip of thickness 55 nm was deposited onto a 5 mm × 5 mm × 0.5 mm BK7 glass substrate with the aid of an e-beam evaporation machine. A 1550 nm light source operating over the telecommunications C-band is launched into the input optical fiber port and routed to a fiber collimator through an optical circulator. This optical signal is then collimated by a collimating lens integrated onto the end of the optical fiber.

A side of the micro-prism is glued onto the collimating lens and another side is coated with (i) a light reflector and (ii) a silver nano-thin-film SPR sensing layer (deposited on a glass substrate to form an SPR chip). The collimated optical signal emerging from the fiber collimating lens strikes the SPR sensor chip at a total-internal-reflection (TIR) angle of θ (see Fig. 1). After reflection back off the Ag coated side, the beam strikes the SPR sensor chip again at the same TIR angle, θ, and eventually couples back through the collimating lens into the optical fiber, where a 3-port polarisation maintaining single-mode optical fiber circulator routes it to a Newport 1830-C optical power meter, employing a Newport 818-IR free-space photo-detector, for analysis.

When the sensing Ag layer is exposed to an aqueous solution, the light source launched into the micro-prism produces an evanescent field that excites a surface plasmon wave at the metal-prism interface, thus resulting in optical signal attenuation.

The coupling of the evanescent optical field into the generated surface plasmon wave strongly depends on the wavelength of the input optical signal, the water refractive index, the ambient temperature and the metallic layer properties (thickness, pattern, etc.). The spectral reflectance of the SPR sensor is typically evaluated by an optical spectrum analyser (OSA) at the monitoring end.

Compared to other fiber-optic based SPR sensors, the proposed one has many advantages. First, it uses disposable sensing chips so that it can be used as a platform for remotely sensing and measuring various compounds using different sensing chips. Second, conventional fiber optic SPR sensors have a limited measurement range, while the proposed sensor has a wide and selectable measurement range that depends on the selected chip substrate material. By increasing the refractive index of the substrate a wider measurement range can be achieved. Third, the sensitivity of the proposed sensor depends on the refractive index of the selected substrate of the sensor chip as well as the wavelength of the light source, therefore, the sensitivity of the sensor can be much higher than that of a conventional fiber-optic sensor counterparts, whose sensitivities are limited by the properties of the optical fiber used. These unique properties of the proposed SPR sensor are especially crucial for various emerging sensing applications, such as toxic gas detection, food quality and safety analysis, medical diagnostics, and environmental monitoring.

3. Theoretical analysis

In a conventional Kretschmann-type SPR sensor configuration, the incident light interacts once with the surface plasmon wave at the interface of the metallic nano-thin-film layer and the sensed sample (this is referred to as “single pass configuration”). Since the thickness of the sensor chip substrate is far greater than the light wavelength, a three-layer Fresnel equation for the p-polarized light can be used for the reflected light intensity R [14]

\[
R = \left| \frac{r_{pm} + r_{ms} e^{2jk_{pm}dn_{m} \delta n}}{1 + r_{pm}r_{ms} e^{2jk_{pm}dn_{m} \delta n}} \right|^2
\]

where \( r_{pm} \) and \( r_{ms} \) are the reflection coefficients for the substrate-metal layer interface and the metal layer-sensing sample interface, respectively. \( r_{pm} \) and \( r_{ms} \) are given by [14]

\[
r_{pm} = \frac{k_{pm} - k_{m}}{k_{pm} + k_{m}}
\]

\[
r_{ms} = \frac{k_{ms} - k_{s}}{k_{ms} + k_{s}}
\]

where

\[
k_{j} = \frac{\omega}{c} \sqrt{\varepsilon_{j} - \varepsilon_{p} \sin(\theta)^2}
\]

\[
\varepsilon_{j} = \text{the dielectric constant of the medium } j (p \text{ for prism, } m \text{ for metal and } s \text{ for sample media}),
\]

\[
k_{j} = \text{the wave vector perpendicular to the interface in medium } j,
\]

\[
\delta n = \text{the thickness of the metal layer},
\]

\[
\theta = \text{the angular frequency of the incident light},
\]

\[
\omega = \text{the light incidence angle at the substrate-metal interface},
\]

\[
c = \text{the speed of light}.
\]

Unlike the conventional Kretschmann configuration, the proposed fiber optic SPR sensor structure enables the optical beam to strike the metal-sample interface twice. Since the two reflections at the interface are identical, the light intensity coupled back to the fiber collimator \( \mathcal{R} \) can be expressed as,

\[
\mathcal{R} = R^2
\]

To compare the performances of the conventional single-pass configuration and the proposed SPR sensors, a numerical simulation was conducted. Fig. 2 shows the calculated SPR spectra for sample refractive indices of 1.33 and 1.3305, and different silver SPR thin film thicknesses of (a) 45 nm, (b) 50 nm and (c) 55 nm. In each case, the full width at half maximum (FWHM) bandwidth for the double-pass configuration is wider than that for the single-pass configuration. Compared to the single-pass configuration, the double-pass configuration exhibits a wider FWHM bandwidth, as evidenced from Figs. 2(a–c). It is important to note that for an SPR sensor based on wavelength interrogation, the double-pass configuration experiences a lower signal-to-noise ratio, thus less detection accuracy compared to the single-pass configuration, while sharing the same sensitivity (wavelength shift for a given index change). In addition, as shown in Fig. 2(c), for the double-pass configuration, a relatively thicker SPR chip metal layer results in a relatively narrower FWHM bandwidth.

On the other hand, if the intensity interrogation is used (i.e., the source wavelength is fixed), the double-pass configuration could exhibit better sensitivity than that of the single-pass configuration.
For example, if the normalized reflected light intensities $R_a$ and $R_b$ for solution refractive indices 1.3305 and 1.33, respectively, then the change in reflected intensity for the single-pass configuration is $R_a - R_b$, while for the double-pass configuration the change in reflected light is

$$R_a^2 - R_b^2 = (R_a - R_b) \times (R_a + R_b).$$

We define the figure of merit as

$$\text{FOM} = R_a + R_b.$$  \hfill (7)

The FOM indicates the improvement in double-pass configuration sensitivity in comparison with the single-pass configuration. For example, for a silver SPR layer thickness of 45 nm (Fig. 2(a)), the FOM at the wavelength used for intensity interrogation (solid vertical line) is almost 1.0 for solution refractive indices of 1.3305 and 1.33. On the other hand, for a silver SPR layer thickness of 55 nm the FOM is greater than 1.0. It is useful to mention that the FOM is always greater than 1.0 for the double-pass configuration when the thickness of the silver SPR layer reaches 55 nm, as shown in Fig. 2(c). Therefore, in an intensity interrogation based double-pass configuration, it is always better to use a relatively thick SPR metal layer to ensure that the FOR is greater than 1.0.

4. Experiments and discussions

Fig. 3 illustrates the experimental setup for water salinity measurement, which was aimed to demonstrate the concept of the proposed fiber optic SPR sensor. The fiber-optic SPR sensor comprised a fiber-optic polarisation maintaining (PM) collimator, a micro-prism, a high-reflection mirror, and a detachable Ag nano-thin-film SPR sensing chip, as illustrated in Fig. 3(b).

The custom-made micro-prism had an isosceles-triangular base of angles 53°–63.5°–63.5° (corresponding to a base size of 5 mm × 5 mm × 4.46 mm) and 5 mm height. The fiber collimator was attached to one of the 5-mm sides, and the high-reflection mirror was attached to the other 5-mm side, while the Ag nanothin-film SPR sensing chip was attached onto the 4.6-mm side of the prism (this side is typically left uncovered so that SPR chips of different properties can be either removed and/or attached). A photograph of the developed fiber-optic sensor demonstrator is shown in Fig. 3(c). A detachable SPR chip with 55 nm Ag nano-thin-film, deposited onto a BK7 glass substrate using an e-beam evaporator system, was adhered to the sensor with an index-matching optical epoxy. A 1550 nm optical signal was launched into the input port of the sensor, and the reflected light coupled by the optical fiber PM collimator was routed, through a circulator, to a free-space photo-detector (Newport 818-IR) for power measurement.

In the experiments, the refractive index was varied by dissolving NaCl into 500 mL of pure water, in which the sensor was immersed. Fig. 4 shows the reflected optical power versus the salt concentration, for an interrogation wavelength of 1550 nm. It is obvious from Fig. 4 that the reflected power increases with increasing the salt concentration. Note that, for changes in salt concentration between 0 ppm and 3000 ppm and between 8000 ppm and 11,500 ppm, the reflected optical power changes slower than that corresponding to the range 3000–8000 ppm. Note that the linear response range between 3000 ppm and 8000 ppm depends on the interrogation wavelength. For example, using a long wavelength shifts the linear response range towards the low salt concentration range. By fitting the measured data of Fig. 4 with a straight line, an average sensitivity of 4.8 μW/ppm is calculated. The sensitivity of the proposed fiber optic sensor is 3.672 × 10⁻⁴ RIU. This value was calculated using a table showing the refractive indices of sodium chloride (NaCl), different salinities at room temperature [15]. The affinity...
The binding capability of the SPR sensor for polysaccharides (namely, xanthan gum) has been demonstrated and reported in [16].

The noise floor of the optical power meter was approximately 0.01 µW (optical power). The smallest change in the refractive index that could be measured confidently was 3.672 × 10⁻⁵ RIU, which corresponds to a signal-to-noise ratio (SNR) in excess of 10 dB. The results shown in Fig. 4 demonstrate the principle of the proposed fiber optic SPR sensor.

5. Conclusion

A double-pass fiber optic SPR sensor, comprising a fiber optic collimator, a micro-prism, a high-reflection mirror and a re-attachable silver nano-thin-film SPR sensing chip, has been proposed and its concept has experimentally been demonstrated. Experimental results have demonstrated a linear response versus salt concentration with a sensitivity of 4.8 µW/ppt over a wide salinity range. The developed sensor demonstrator has been packaged for water-proof operation, demonstrating excellent flexibility and robustness when immersed in aqueous solutions. The sensor will have applications in biology, chemistry, medical, environment, food, toxic gas, and security.

Acknowledgement

The authors acknowledge the financial support of the National Centre of Excellence in Desalination Australia which is funded by the Australian Government through the National Urban Water and Desalination Plan.

References


Biographies

David Michel was born in the U.K. He received the B.Sc. in computer science and B.Eng. telecommunications from Edith Cowan University, Western Australia in 2009 and a M.Sc. in engineering from Edith Cowan University, Western Australia in 2012. Currently, he is completing a Ph.D. in philosophy with the research topic of fiber optic based Surface Plasmon Resonance sensors in Edith Cowan University, Western Australia.

Feng Xiao was born in Jiangxi Province, P.R China, in 1975. He received the B.S. degree in applied physics from Nanchang University in 1998, and the M.S. and Ph.D degrees in electronics engineering from Peking University in 2003 and in 2007, respectively. From 2007–2015, he was a Research Fellow with the Electron Science Research Institute, Edith Cowan University, Australia. He is the author of two book chapters, more than 90 articles, and more than 3 inventions. His research interests include optic devices, fiber-optic sensors, optical communication, Surface Plasmon Resonance, plasmonic devices, optical phased array antenna, and microwave photonics. He is an editorial board member of the Austen Journal of Biosensors and Bioelectronics, and holds two patents.

Kamal Alameh received the M.Eng. degree in photonics from the University of Melbourne, Melbourne, Australia, in 1989, and the Ph.D. degree in photonics from the University of Sydney, Sydney, Australia, in 1993. He is currently a Professor of Micro-Photonics and Director of the Electron Science Research Institute (ESRI), Edith Cowan University, Joondalup, Australia. He is also Adjunct Professor with the Department of Information and Communications, Guangji Institute of Science and Technology (GIST), Korea and South Wales University, Wales, U.K., and Guest Professor with Southeast University, Nanjing, China. He has pioneered the integration of microelectronic and photonics sciences and developed a new and practical research area, “MicroPhotonics,” and currently he is engaged in research and development on Opto-VLSI, optoelectronics, and micro-/nano-photonics targeting innovative solutions to fundamental issues in ICT, agriculture, health, energy, consumer electronics, and security and defence. He has authored or co-authored over 350 peer-reviewed journal and conference papers, including three book chapters, and filed 28 patents.