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REVIEW

Optical Sensors based on Surface Plasmon Resonance

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This brief review presents the recent process in optical sensors based on surface plasmon resonance (SPR). In particular, it will focus on the optical sensors that employ the change of refractive index as the sensing transduction signal. Various detection schemes of optical sensors which include phase modulation, wavelength modulation and intensity modulation are discussed. The performance advantageous and disadvantageous of the description of optical sensors structure and their respective experimental configurations are also described. The examples of detection in chemistry, biology and heavy metals will be presented. Future prospects of surface plasmon resonance (SPR) sensing technology is also discussed.

Keywords: Surface plasmon resonance, Sensitivity, Sensor, Detection limit, Prism, Optical fiber.

INTRODUCTION

Under certain condition, optical beam can be used to excite the surface plasmon wave along the interface between metal and dielectric. Optical sensors based on surface plasmon resonance (SPR) have various applications in chemistry, biology and environment. In 1968, Otto has firstly investigated the surface plasmon resonance [1]. Similarly, Orfanisdis [2] demonstrated an optical excitation of surface plasmon waves by using different configurations of attenuated total reflection method. Moreover, Pitarke *et al.* [3] have reported the theoretical SPR wave, which is sufficient for experimental works. Based on SPR phenomena, there are various optical sensors have been made for different purposes such as gas detection [4], biological detection [5], chemical detection [6] and temperature detection [7].

Nowadays, there are numerous SPR sensors based on the phase detection, wavelength detection and intensity detection. Each scheme has not only different emphasis but also different advantageous and disadvantageous. Table-1 shows that the achievement of SPR sensors based on distinctive phase modulation, intensity modulation and wavelength modulation. The result shows that the phase detection is one of the promising parameters in SPR sensors for the sensitivity of the materials.

TABLE-1 COMPARISON OF THE DETECTION LIMIT OF VARIOUS SCHEMES IN SPR SENSORS

SPR schemes	Intensity modulation	Wavelength modulation	Phase modulation
Detection limit (RIU)	6.4×10^{-6}	3×10^{-7}	5.5×10^{-8}

Recently, there are some articles [8-10] and books [11,12], which have summarized the several applications of SPR sensors. To make that more understandable, this review reported various SPR detection schemes. Moreover, the review also introduces the recent wok on the developing and applications of SPR sensors in biological, chemical and heavy metals analysis.

Surface plasmon resonance

Excitation of surface plasmon: The optical sensors based on the surface plasmon resonance are imitated from two main configurations. Firstly, in Kretschmann-Raether configuration, a thin metal layer is sandwiched between the prism and the air. Otherwise, the Otto configuration, there is a gap between the metal and the prism. However, the Kretschmann-Raether geometry is more conventional scheme to generate the surface plasmon wave in sensing technique [13]. The incident light wave is also transverse magnetic wave that can be existed on the metal

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air interface and that can be used to excite the surface plasmon under the boundary condition [14].

Most of optical excitation of surface plasmon is consist of prism [15], waveguide [16], optical fiber [17] and grating [18]. The surface plasmon resonance wave is described as an electromagnetic wave propagating along the interface between metal and dielectric medium. To explain how the surface plasmon occurs, an analysis of the electromagnetic properties of an interface is necessary. For three layer system, glass/metallic/analyzed solution, the propagation constant (β) of the surface plasmon wave propagated along the metal-analyze solution interface and the angular frequency, which can be described as follows:

$$\beta = \frac{\omega}{c} \left(\frac{\varepsilon_{\text{bm}} \varepsilon_{\text{s}}}{\varepsilon_{\text{bm}} + \varepsilon_{\text{s}}} \right)^{1/2} \tag{1}$$

where ε_{bm} and ε_{s} are the dielectric constants of the metal and the analyze solution, ω is the angular frequency of the incident light and c is the speed of light. In the prism coupling configuration (Fig. 1), an incident light wave passes through a high refractive index prism and then the light is totally reflected at the prism-metal interface. The evanescent wave will generate and penetrate into the thin metal film. The propag-ation constant of the evanescent wave can be calculated as follows:

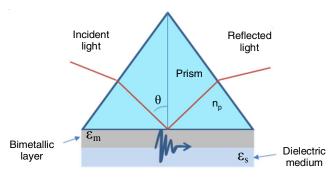


Fig. 1. Schematic of the SPR sensor based on prism

$$k_{x} = \frac{\omega}{c} n_{p} \sin \theta \tag{2}$$

where θ is the incident angle of the light, n_p is the refractive index of the prism, c is the speed of light and ω is the angular frequency of the incident light. By given the proper thickness of metal film and the resonance angle of light, the propagation constant of the evanescent wave match that of the surface plasmon [19] (eqn. 3):

$$k_{x} = \frac{\omega}{c} n_{p} \sin \theta_{res} = \frac{\omega}{c} \left(\frac{\varepsilon_{bm} \varepsilon_{s}}{\varepsilon_{hm} + \varepsilon_{s}} \right)^{1/2}$$
 (3)

Optical sensors based on the prism coupling is the most convenient in SPR geometry and normally has the best detection limit. However, the prism is enormous and not easy to integrate. A good anternative to the prism scheme is waveguide coupling, which offers more accurate and robust integratation. The light wave joins into the waveguide through metal-dielectric interface and generate the evanescent wave penetrating to the metal

film. The excitation of suface plasmon of the waveguide is in the same way with the prism configuration [16,20].

Additionally, the fiber has also been used in optical sensor [21]. The cladding of optical fiber is removed and then deposited with the metal thin film. The excitement of surface plasmon wave in the fiber is an easy and flexible way. Many different optical fibers has been used to develop the SPR sensor such as single mode optics fiber [22], step-index fiber [23], single mode polarization maintaining optical fiber [24], multimode optical fiber [25] and tapered fiber [26].

Moreover, the surface plasmon wave can be excited by the grating structure, which can be one of the best performant in optical sensing [27]. The optical excitation of surface plasmon is accompanied by the transfer of the light wave energy into the surface plasmons. In this way, the light wave is incident on the metallic grating, which has the period of groves. The diffraction grating scatters the incident light wave at the surface of metal grating. After the reflection of incident light into the dielectric medium, the wavenumber of the mth diffracted evanescence wave is given by the eqn. 4 [28,29]:

$$k_x = \frac{\omega}{c} n_d \sin \theta + mW = \frac{\omega}{c} n_d \sin \theta + m \frac{2\pi}{\Lambda}$$
 (4)

where n_d is the diectric refractive index, m is the diffraction oder (m = 0, ±1, ±2, ±3, ±4,.....), Λ is an period of the grating. When m-th diffraction is coupled to the suface plasmon, the coupling condition can be expressed as:

$$k_{x} = \beta \Leftrightarrow \frac{\omega}{c} \left(\frac{\varepsilon_{bm} \varepsilon_{s}}{\varepsilon_{bm} + \varepsilon_{s}} \right)^{1/2} = \frac{\omega}{c} n_{d} \sin \theta_{res} + m \frac{2\pi}{\Lambda}$$
 (5)

where θ_{res} is the resonance angle of incident light. The reflectivity dip of the reflected light is indicated to measure the analyzed solution in this grating structure, which has not only high resolution but also good sensitivity.

Choice of metals: The excitation of surface plasmon wave is occurred on the metal-dielectric surface. To obtain the good sensitivity and signal to noise ratio, the applicable metal is very important. There are several kinds of metals, which are gold, copper, aluminum and silver, can be used in the optical sensors. In this case, copper has the unstable properties and easily to get an oxidation. The best performance of optical sensor is generated by gold, which has the stable chemically and mechanically during the experiments [30]. Two kinds of structures of gold have usually been used to generate the surface plasmon wave. Firstly, the spherical gold nanopartical has already considered applying in the optical fiber sensor [31]. The sensitivity of the sensor is depended on the particles size, interparticle distance and particle alignment. Secondly, the other gold structure is the thin film based on the prism coupling and optical fiber [30,32]. In this case, the resolution of the sensor is more accuracy which also depend on the thickness of gold layer.

In other case, the silver thin film has been reported, which offers attractive advantages in the sensing [33]. However, silver is easy to get oxidation when it emerges into the dielectric constant. To overcome this problem, the combination of these metals is required. Many researches presented the performance

of the bimetallic thin films [34-39]. There are several combinations such as Au-Ag [34], Ag-Au [36], Au-Al [38] and Cu-Al, Ag-Cu, Cu-Au [39]. Moreover, the combination of bimetallic layer not only displays the resolution but also gives the stability in the sensing performance. In the bimetallic combination, the sensitivity is also strongly depended on the thickness ratio of the inner and outer layer.

Additionally, the application of optical sensor based on the metal and bimetallic layer is not convention to apply in the biological sensing because of the interaction between such metals and biomolecule is too poor. In other to improve the sensitivity and the stability of biosensor, graphene is a suitable candidate material to replay the metal [40]. The special property of grapheme due to the surface to volume ratio is so high that is efficient absorbent of biomolecules. The study of absorbance of biomolecules of biosensor based on graphene deposition is excited in the future work.

Resolution and sensitivity: For best forming sensor, two important factors *i.e.* sensitivity and resolution should be high as possible. Sensitivity is an important parameter to estimate the sensor performance. Sensor sensitivity (S) is defined as a ratio of the output signal change dU (intensity change, phase shift or resonance wavelength shift) to the change of refractive index of the analyze solution dn (refractive index unit (RIU)) [23].

$$S = \frac{dU}{dn}$$
 (6)

Sensor resolution is another important parameter to characterize the sensor performance. It refers to the smallest change in refractive index of the analyze medium that produce the detectable change in the output signal [41]. The detection limit (DL) of SPR sensor is also related to the sensor resolution. The limit of detection has usually been indications as the concentration of analyze solution that generates the output signal corresponding to three times standard deviations of output measured by the blank sample [42]. In the optical label free sensors, there are three approaches to classify the detection limit. Firstly, detection limit in the unit of refractive index (RIU), which is not only approved unit in the sensing of the refractive index change in the bulk solution but also common unit in the SPR sensors. Secondly, the surface mass density in term of pg/mm², which usually uses in the biosensor to determine the binding between biomolecule and sensor surface. Finally, the unit is in the concentration of sample in term of ng/mL. There is a relationship among them and it needs to specify the detection limit in each of units for each different biosensor.

SPR sensing classification

Phase measurement: The phase measurement, a common technique in the SPR sensing, is generated on the interference between a signal beam and a reference beam by the photodetector. Optical heterodyne is a conventional technique used to detect the phase in SPR sensors. In this technique, two laser beams with slightly different frequencies are emitted and made an interference with each other. The signal is called the beating signal, which is strongly depended on the different frequency between two beams.

Nelson et al. [43] reported the phase measurement based the heterodyne detection. They reported the acousto-optic modulator (AOM) used to generate two beams with difference frequency (140 MHz). These beams are recombined by a polarizing beam splitter as a result of reference and signal path. In the reference path, the two beams are recombined with a 45° polarizer and joined into the photo-detector. In the signal path, the optical beam is coupled into the SRP cell to generate the phase shift between p- and s-polarization then pass through the low pass filter to achieve the beating frequency of 140 MHz. To compare with standard phase measurement, both of reference and signal paths are mixed with the second signal, which is generated by the function generator. And the both of them are low pass filter again to cancel out higher harnomic. A phase detection circuit is candidate to detect the SPR phase shift between the reference and signal path. The phase detection based on the heterodyne detection is accurate and sensitive. The phase resolution depend on the thickness of the metal, its optimum value is 53 nm of the silver in their work. This proposed method can be measured small change of refractive index unit of glycerol solution (5 \times 10⁻⁷ RIU). The result matches with the theoretical prediction and achieved high resolution. However, this method also has some limitations, which are due to the experiment setup and expensive instruments.

In 2003, Wu et al. [44] also present a new phase detection method based on heterodyne interferometer. In their work, a He-Ne laser with 633 nm wavelength was used. The optical beam splits into two paths by polarizing beam splitter (PBS-1). The two AOM are used to excite these beam with slightly different frequency (60 kHz). Then two optical beams are recombined by the other polarizing beam splitter (PBS-2). After the PBS-2, the light is again conveyed into two beams by a beam splitter. Each of optical patches contains the p- and s-polarization component. One of the beams as the reference beam is got the interference after passing through the polarizer and receive by the photo-detector. In the other part, the light is joined into the SPR sample, which is made of Bk7 glass prism with gold film coating. Under the SPR condition, the reflected beams pass though the polarizer and converge into the electrical signal by the photo-detector. The phase difference between signal path and optical path is detected by the commercial phase detector. The refractive index resolution can be achieved is 2×10^{-7} (RIU), which is significant better than Nelson's work.

Wu *et al.* [32] also presented the new scheme of phase detection based on Mach-Zehnder interferometer [32]. The experimental setup is easier than previous works. In this work, the optical beam is directly jointed into the SPR sensor after splitting by beam splitter. In the referent beam, the piezoelectric transducer (PZT) with period movement is indicated to reduce the environmental noise and obtain the best signal. After recombination of the referent and signal beam, the optical beam is passed though the Wollaston prism to separate into pand s-polarization component. The phase is measured by detector and digital storage oscilloscope. In their experiment, the optimal sensitivity limit is 5.5×10^{-8} (RIU) for glycerolwater mixing case. Moreover, they also used the biomolecular

their measurement. The estimation of sensitivity for BSA antibody was 7.4 ng/mL.

Naraoka and Kajikawa [45] introduced a SPR induce phase shift by rotating analyzer method. The single optical beam was used to excite the SPR and the pass through the analyzer (Gra-Tompson prism). In this case, the intensity of the reflected beam was detected and then the phase shift between *s*-and *p*-component was extracted by using the fitting profile. In their work, the index sensitivity was the order of 10⁻⁷ (RIU), which is not significant better than heterodyne detection. However, their work is simple the optical geometry and promise to apply in the spectroscopic analysis.

Lee *et al* [46] enhanced the sensitivity of the SPR sensor using the wavelength modulation method. In their case, the experimental configuration is based on the Michelson interferometer, where the output light of the Michelson's setup is an incident light to excite the SPR wave. In their operation, the heterodyne light source is used. The reflected light of the sample is split into two parts by beam splitter. In each of optical path, the p- and s-polarization interfere with each other depending on the polarizer. The phase shift is detected by commercial phase detector. The experiment result reports the index resolution was 3×10^{-7} (RIU). Although the result is not greater than the other, but their scheme have several advantages such as simple setup, stability and real-time monitoring.

Resonance wavelength measurement: SPR sensors based on the phase measurement are due to interferometer, which is easy to get variation of signal as discussed above. To overcome this problem, a simple detection method is reported, where a heterodyne light is passed through the SPR sensors. Then the output light will analyze by the spectrum analyzer. Several scientists [47-51] had performed the theoretical works to investigate the feasibility and optimize conditions for SPR sensors.

A theoretical investigation on the wavelength domain was performed by Homol and Yee [14]. In this work, a thin glass slide (light pipe) with a metal deposition is used as the sensing element. For resonance wavelength measurement, the white light is passed into the light pipe. The SPR conditions are strongly depended on the incident angle and wavelength modulation. When the refractive index of the sensing medium is changed, the reflectivity will be arbitrated as a function of wavelength. In this work, theoretical analysis has carried out the optimal parameters, which have a new configuration, the wavelength range (500-1000 nm) and the sensing region length of the light pipe. These advantages could be proposed to many applications including chemical analysis.

Tubb *et al.* [52] have reported a new scheme of SPR single mode optical fiber. In this work, a single mode fiber has used to make optical sensor, whose diameter was exponential, from the fiber cladding diameter of 125 μ m down to about 3 μ m of core diameter. The sensing region will be deposited the metal (silver or gold) by thermal or electron beam evaporator. The halogen source is launched into the optical fiber sensor and then the output light will be analyzed by a Stanford SR530 lock-in amplifier. The resonance wavelength has been measured base on the refractive index change of methanol (n = 1.326), acetone (n = 1.357) and propanol (n = 1.375). The sensitivity

was 5×10^{-4} RIU which is being detectable. This method, it is easy to fabricate the sensor and applied successful as an immunosensor and biological sensors.

The resolution of optical sensor based on SRP resonance wavelength measurement is significantly improved by Iga et al. [33]. In this setup, a multimode GI fiber with 50 µm core diameter was used, then 10 mm sensing length of single mode SI fiber segment (3.1 μm core diameters) will be inserted into the multimode fiber by thermal fusion splicer (FSM-40F, Fujikura Ltd.). The silver deposition (50 nm) in the sensing region is coated by using a FR sputtering machine, which is especially design to construct the symmetrical deposition on the sensing surface. In this setup, the halogen lamp was also used as a light source, which is joined into the transmission line fiber. The spectrum of the output signal is detected by spectrum analyzer and the sensitivity achieved 1.5×10^{-4} RIU. This group has also used the same fiber structure with gold deposition [30]. In the case of gold sensing film, the sensing resolution was 3.64×10^{-5} RIU, which is 12 times better than the case of silver film. Even though the sensitivity of the fiber optical sensor based on the resonance wavelength measurement is not higher than the phase detection, it has several advantages such as simple setup, easy to fabricate the sample and low cost.

Intensity measurement: Beside the phase and resonance wavelength detection, intensity can also be used to extract the information of SPR sensor sufficiently. Intensity method is simpler than two other methods as mentioned above. In this method, the monochromatic light is passed through the sample and then the reflected light will be recorded by power meter. In the intensity measurement, fibers are indicated as a beneficial material for SPR optical sensor fabrication because they are cheap materials and convenient for the signal propagation. Slavik et al. [53] reported the sensing range of the fiber sensor is 2 cm by using optical fiber. The sensing region is coated with different materials such as aluminum, tantalum pentoxide, gold, etc.. The thickness of each layer was controlled to achieve the optimum resolution and the best resolution can be obtained 4×10^{-5} RIU. Iga *et al.* [33] have represented their work by using the hetero-core fiber structure with gold thickness dependence. That group also used the optical fiber structure to detect the reflected light amplitude.

Moreover, the D-type of single mode fiber was also used for optical sensor design [54] consisted of 4 mm long of the sensor length deposited with 1 nm SiO₂ and different Au thickness as an outer layer. The He-Ne laser is the light source, which is passed through the polarizer and the joint into the fiber sensor by the objective lens. The best resolution was achieved at 2×10^{-5} RIU, which is significantly better than the other group.

Recently, a new approach for nanohole rectangular array SPR optical sensors based on intensity modulation has been developed by coordinating the SPR effect and micro or nano array sensor [55-57]. Blanchard-Dionne *et al.* [58] reported the nanohole rectangular array fabricated with various structural size [58]. To generate the SPR effect, the Au film with 100 nm of the thickness was used. When the light pass through the sensor, the output beam split into two paths by using Wollaston prism. The different concentrations of ethanol were

used as an analyte. The detection resolution achieved 6.4×10⁻⁶ RIU, which is much better than the other works. Although the SPR sensor based on intensity modulation does not give higher resolution, however, has some advantages such as simple setup, low cost and real time monitoring. Furthermore, SPR nanohole array is a new approach, which is sufficient for multi-detection.

Majors areas of applications of SPR sensors

and a promising in chemical detection. To avoid the variation of the analyte and enhance the sensitivity of chemical SPR sensors, the metal film to be modified with specific chemical process, which is dependent on the gas used. Most of the chemical SPR sensors, the SPR signal detects based on the change of the refractive index of the sensing gas, which either

The optical sensors based on the SPR are simple, sensitive

Chemical sensing based on surface plasmon resonance:

change of the refractive index of the sensing gas, which either occured in the chemical reaction or an absorption of the analyte with the sensing surface. The early applications of SPR sensors were used to monitor the change of concentration of acetone [59], methanol, isopropanol and hexane vapours [60], measured the refractive index change of chlorinated and aromatic compounds [61], detection of the cycle replacements of Ar and N₂ [62], temperature measurements [7,63], distinguish specific gases [64]. To measure the change of refractive index of the transducing medium by the absorption of sensing surface, there are several articles related to the selective gas detection, which include the change of concentration of vapors of methanol, alcohol, ketone and aldehyde by absorption in polyethylene

glycohol film [4], monitoring the change of NO₂ gas by porp-

hyrin Langmuir-Blodgett films [65] and 18-crown-6 H₂pc-

phathalocyanine films based on Au on Si grating structure [66]. **Biological sensors based on SPR:** The SPR sensors have also been developed for measuring the characteristics of biomolecules. The SPR sensors for examination the interaction of two biomolecules can be classified into five steps: (i) incubate or capture the first targets, (ii) inject the different concentration of the second binding targets, (iii) perform the data and (iv) regenerate the sensor. Several types of biomolecule elements used in SPR biosensors are nucleic acid, antigen-antibody and proteins. There are numerous scopes in SPR biosensors such as immunology in searching the antigens-antibody, chaperones to study the interaction between protein and protein, screening for new target biological molecule to understand the gene expression's mechanisms and signal transduction for different binding interactions [67]. The detection methods in a particular measurements is strongly depended on the size, concentration and binding characteristic of targets [10].

Recently, SPR has been used to investigate the absorption and desorption of IgG on the sensing surface with protein A modification [52], monitoring the effect of glucose, lactate and human IgG [68], self-assembled using anti-human ferritin monoclonal antibodies for specific binding of antigens [69], to measure the immobilization of PSA-ACT mAb and PSA-ATC complex on the PSA sensor surface [70], displays the detection of different concentration of fibrinogen [71]. In order to enhance the absorption of biomelecule or enzyme, the SPR sensing surface is modified by thin polymer layer [72,73].

Moreover, the covalent binding between antigen and antibody has also proved a powerful method for biosensors [74-78]. Another important feature is study an immobilization of tTGas related to the Ca²⁺ concentration [79].

Heavy metals sensors based on SPR: The heavy metal sensors based on SPR have high sensitive and selective detection of Pt²⁺ [80,81], K⁺ [82], Cd²⁺, Zn²⁺ and Ni²⁺ [83,84], Pb²⁺ [85], ultrasensitive detection of Hg²⁺ ions [86,87], *etc*.

Future applications: The SPR sensor SPR sensor based on prism, optical fiber, waveguide and grating are now become commercial. However, now researches based on photonics crystal and ring resonator, even though beginning, may have considerable potential for next generations [88,89]. Finally, the SPR sensors should have mobile analytical systems. The integration of light source, SPR sensor platform and detector on board or on chip with optical and electrical control are the other factors which is to be considered. Moreover, the integration between SPR sensor platform and microfluidic devices is also a great potential to minimize the volume injections for biosensors applications and multi-detection analysis of metals, gases and compounds.

Conclusion

In this review, recent process in various optical sensors structures and their detection scheme for label free detection are reviewed. The collaboration of different metals and receptor has carried a lot of enhancement in sensing of various chemical, physical, biological and environmental applications. It is believed that this review will help enough valuable information for researchers and benefitted them to go further exploration and experimentation in this area.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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