Transmission and Reflection SPR Disposable Fibre Probes for Bio-chemical Sensing

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***Abstract*— A comparison between the characteristics of transmission and of reflection based surface plasmon resonance (SPR) fiber sensors is presented. Both sensors are built using a multimode fiber with 400 µm silica core diameter, un-cladded on a length of 2 cm and then coated with a 50 nm gold layer. The reflection based setup uses a bifurcated fiber bundle to avoid complex and expensive circulators or couplers. The effect of misalignments and mating tolerances are investigated. The comparison between transmission and reflection based setups shows that the same fiber sensor exhibits a higher sensitivity when used in the reflection based configuration. The latter is also advantageous since it allows the use of the fiber sensor as a disposable probe.**

Keywords—; Fibre Optic Sensors; Bio-chemical Sensors; Surface Plasmon Resonance (SPR).

I. INTRODUCTION

Surface plasmon resonance (SPR) is one of the most used optical sensing techniques for the detection and monitoring of chemicals, and SPR based-sensors have been widely employed over the past two decades for label-free, real-time analyses of bio-chemicals exploiting antibody-antigen reactions [1]. Actually, SPR prism-based setups have already been turned into commercial products, so research is now mainly focused on fiber optic configurations since these leads to more compact - particularly small form factor and minimally invasive probes - and cost-effective sensing systems [2-3].

SPR occurs at the interface between a thin metal layer and a dielectric; gold is commonly used as the metal for its chemical inertness. The interrogation of fiber SPR sensors is typically done using a broadband source and a spectrometer to evaluate the intensity and spectral position of the light components absorbed due to the excitation of the surface plasmon wave, which in turns depends on the refractive index of the material in contact with the metal layer. The drawback of such approach is that parasitic fluctuations in the measured signal amplitude are indistinguishable from actual refractive index variations. This is especially critical in long-term monitoring applications, where perturbations from the mechanical setup or drifts in the electronic circuits are unavoidable, especially if targeting low-cost solutions. These limitations can be overcome using differential configurations in which a reference sensor can be used to compensate the fluctuations of the measuring sensor [4-5], so they will not be further considered in this paper.

Practical SPR fiber sensors can be implemented in two configurations, which, respectively, make use of transmission or reflection based setups. In the first type, light from the broadband source is launched in the sensing fiber through one of the ends and the spectrum of the transmitted light is recorded at the other end [6-8]. In the second type, broadband light is launched from the same end used to collect the reflected spectrum and the two signals are typically separated by means of a directional coupler.

The main drawback of the transmission based setup is that it requires the sensing fiber to be connected to the source and photodetector through both ends, so that it is difficult to use it as a pinpoint sensor. In general, however, biochemical applications call for real-time, user-friendly probes that can be even used in every-day life. In order to cope with these requirements, a reflection-based arrangement, in which the sensing fiber is connected to the measurement system by one end only, can be beneficial provided the SPR optical fiber sensor can be manufactured as a low-cost disposable probe.

Reflection-based SPR optical fiber sensors have been proposed [9-10], but little emphasis has been put on some typical sensor characteristics (e.g. sensitivity, resolution) and a cross comparison with a transmission-based arrangement is still missing.

In this study, a gold-coated optical fiber has been used to develop transmission and reflection based setup for the measurement of the refractive index of liquids ranging from 1.333 (pure water) to 1.377 (isopropyl alcohol). The effect of mechanical misalignments in the transmission-based setup has been investigated and an all-fiber, alignment-free setup for interrogation of the SPR optical fiber sensor in reflection has been developed using off-the-shelf optical components. Finally, a comparison between the two setups in terms of sensitivity and other features has been investigated.

II. THEORETICAL BACKGROUND

The simplest configuration to understand the surface plasmon resonance (SPR) phenomenon is based on a bulk prism (usually made of glass) whose hypotenuse is covered with a thin metal layer. SPR is a resonant oscillation of electrons that occurs at the metal – bulk dielectric interface, whose properties are strongly dependent on the refractive index of the material in contact with the other side of the metal. SPR is triggered when a properly polarized light is incident at the right angle at the metal – bulk dielectric interface, in such a way that the propagation constant of resultant evanescent wave is equal to that of the surface planar wave (SPW) propagating at the interface between the metal and the bulk dielectric. Similar situation occurs when a thin metal layer is deposited on the core of a slab waveguide or of a fiber. The light reflected into the prism/slab/fiber exhibits a sharp absorption at the SPR resonance wavelength, whose value can be evaluated from the resonance condition [5-6]:

$$k n_c \sin\theta = k\sqrt{\frac{\epsilon_{mr}\, n_s^{\,2}}{\epsilon_{mr} + n_s^{\,2}}} \quad ; \quad k = \frac{2\pi}{\lambda} \qquad (1)$$

The left-hand side term is the propagation constant of the evanescent wave produced as a result of Attenuated Total Reflection (ATR) of the light incident at an angle θ through the material having refractive index n_c. The right-hand term is the SPW propagation constant, with ε_{mr} being the real part of the dielectric constant of the metal film and n_s the refractive index of the dielectric (sensing) layer. According to Eq. (1), the SPR wavelength changes when a change in n_s occurs, and this feature can be exploited to produce optical sensors for refractive index measurements. This analysis, valid for a planar technology, qualitatively holds even in fiber geometry, where the slab is substituted by the fiber core, whereas the sensing layer acts as the cladding of the metal-coated core. The performance of SPR sensors can be quantified by three parameters: sensitivity, signal to-noise ratio (SNR), and resolution [11].

The sensitivity of a SPR sensor is defined as the resonance wavelength shifts ($\delta\lambda_{res}$) versus change in RI (δn_s) of the sensing layer:

$$s_n = \frac{\delta\lambda_{res}}{\delta n_s}\ (nm/RIU) \qquad (2)$$

The signal to noise ratio relates the wavelength shift to the spectral width of the SPR dip, hence defining the accuracy of the sensor:

$$SNR = \frac{\delta\lambda_{res}}{\delta\lambda_{sw}} \qquad (3)$$

where $\delta\lambda_{SW}$ is the full width at half maximum (FWHM) of the SPR curve.

The resolution (ΔN) of the SPR-based optical sensor is the minimum amount of change in refractive index detectable by the sensor. The resolution can be expressed as:

$$\Delta N = \frac{\delta n_s}{\delta\lambda_{res}}\ \delta\lambda_{DR} \qquad (4)$$

where $\delta\lambda_{DR}$ is spectral resolution of the spectrometer used to measure the resonance wavelength. In this work, the focus has been put on the evaluation of the sensitivity.

III. Experimental Section

A. Fabrication of gold-coated SPR sensor and experimental set-up

Following the discussion presented in [12], the fiber chosen for the validation experiments is a 400 µm silica core diameter multi-mode fiber (pure silica core, Tetraethyl orthosilicate – TECS - polymer cladding, 0.39 numerical aperture – NA) to provide mechanical strength, simplicity in handling and ease in mating. A gold layer of nominally 50 nm thickness has been deposited on the exposed core for a length of 2 cm using a RF-assisted plasma sputtering machine. The transmission-based setup is shown schematically in Fig.1, where the source is a broadband SLED (470 nm to 850 nm) and the spectrometer has a resolution of 0.3 nm.

The reflection based setup uses the same fiber, but to simplify the mating while avoiding the use of expensive circulators or couplers, a bifurcated fiber bundle is used to launch the excitation light and receive the signal after reflection (Fig. 2). A mirror has been placed the distal end of the fiber probe.

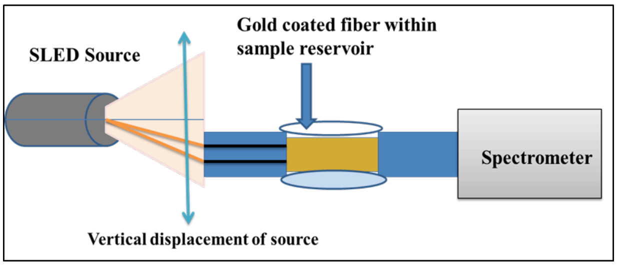


Fig. 1. Transmission based optical fiber SPR sensor set-up with intentional vertical misalignment.

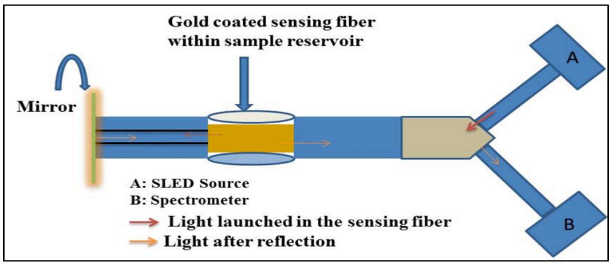


Fig. 2. Reflection based optical fiber SPR sensor set-up.

B. Experimental procedure

In order to analyze the impact of the two probe configurations on the sensor performance, four different solutions prepared with proper mixing of glycerol-water, isopropyl alcohol and pure water have been utilized to provide reference refractive indexes, as summarized in Table 1.

A homemade microfluidic chamber [5] has been realized and used to test the probes in the same working conditions. For each experiment, after injection of the solution under measurement in the chamber, one hundred measurements have been acquired and averaged. The same experiment has been repeated in three cycles, washing the tip with pure water and let it dry before injecting a new solution. The SPR spectra have been fitted with a Lorentzian function to get the accurate minimum position of curve and thus the SPR dip resonance wavelength. Temperature has been kept constant through all experiments to avoid the need for compensation.

TABLE I. SOLUTIONS USED AS THE SENSING LAYER.

	Water-Glycerol	**Concentration Volume %**	**Concentration weight %(20°C)** *(glycerol density 1,2619 (g/ml))*	**Refractive Index (589nm, 20°C)**
1	Pure water	-	-	1.333
2	50 ml-5 ml	9.09 %	11.20 %	1.346 (+$13 \cdot 10^{-3}$ RIU)
3	50 ml-10 ml	16.66 %	20.15 %	1.357 (+ $24 \cdot 10^{-3}$ RIU)
4	Isopropyl alcohol (pure99%)	-	-	1.377

In order to investigate the effect of tolerances in the transmission based setup, an angular misalignment has been intentionally induced by vertically displacing the fiber from the source in steps of 0.1mm using a precision translation stage. For the reflection based setup, where the most critical point is the misalignment between the fiber sensor and the bifurcated junction, tests have been carried out by connecting and disconnecting the sensor to introduce a random un-intentional misalignment and checking the reproducibility of the measures.

IV. RESULTS AND DISCUSSIONS

A. *Transmission based set-up: effect of the source misalignment on the SPR dip*

An example of the typical transmittance of light versus wavelength for a transmission-based setup is shown in Fig 3(a) for three different values of the refractive index of the solution in contact with the metal layer. As expected, there is an evident red shift of the notch wavelength with refractive index increment, as pinpointed in Fig. 3(b). The latter depicts the position of the SPR notch as a function of the refractive index as measured across different experiments. The raw data exhibit a remarkable spread due to the limited reproducibility of the mechanical misalignment, whereas the linear fit highlights the increasing trend.

To further quantify the impact of misalignment, Fig. 4 reports the outcome of the experiment for pure water at different displacements. Clearly, misalignments introduce a decrement in the signal-to-noise ratio of the SPR curve and a non-negligible contribution to the red shift. The SPR dip is visible down to a 0.5 mm misalignment, but in such a condition the shift is ~19 nm, which corresponds to ~10^{-2} RIU, a value unacceptable in most applications.

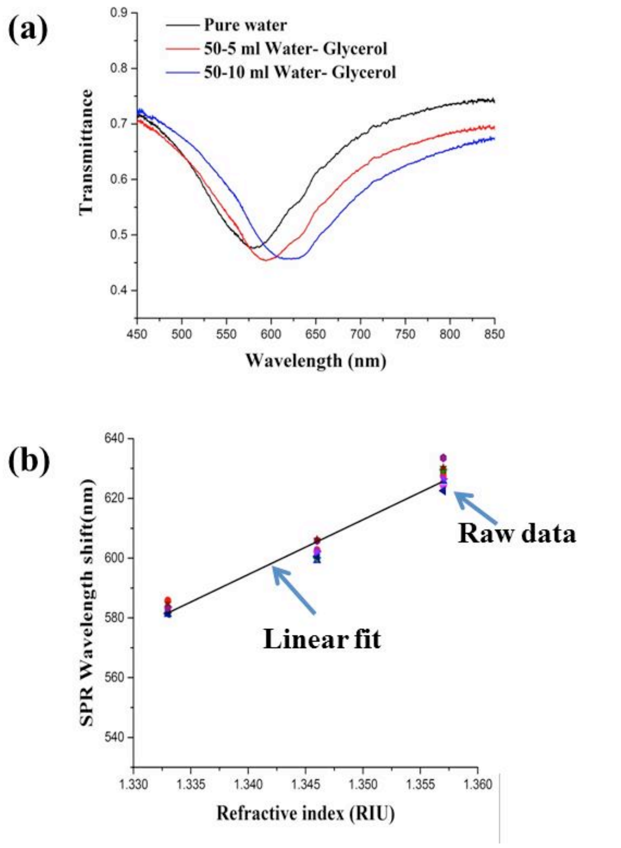


Fig. 3. Characterization of the transmission based setup: (a) example of the spectral transmittance versus wavelength for three cases; (b) notch wavelength position versus refractive index for different cycles of the experiment (symbols) and linear fit (solid line).

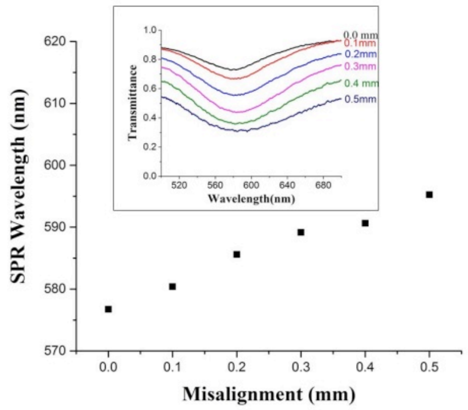


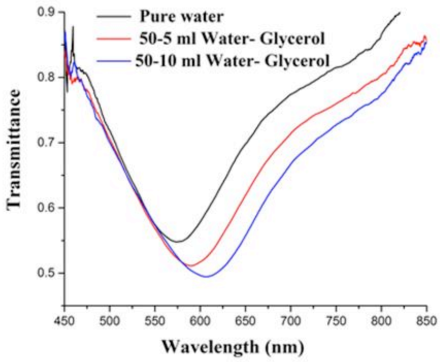
Fig. 4. Analysis of the impact of misalignments in the transmission-based setup: shift of the SPR notch as a function of misalignment (main graph) and raw transmission spectra (inset).

B. *Reflection based set-up: alignment free, improved sensitivity*

The reflectance curves obtained with the same fiber, used in the transmission based setup and here tested in the configuration of Fig. 2 (reflection based setup), are shown in Fig. 5(a). The SPR wavelength shift versus refractive index is then reported in Fig. 5(b), where a remarkable red shift of the

SPR notch can be observed. The SPR shift (mean value) in the case of transmission and reflection based setups have been reported in Fig. 6 for comparison. It can be noticed that the two curves lay at different levels, demonstrating that the reflection based setup exhibits a higher sensitivity than the transmission based one. The calculated sensitivities are 1049.39 nm/RIU and 1172.03 nm/RIU for the transmission and reflection based setup, respectively. The improvement of sensitivity may be due to the fact that light travels twice the sensing region in the case of the reflection-based setup, hence producing a larger interaction length, though this idea does not explain the similar depth of the SPR notch in the two configurations and the phenomenon shall be further investigated.

(a)



(b)

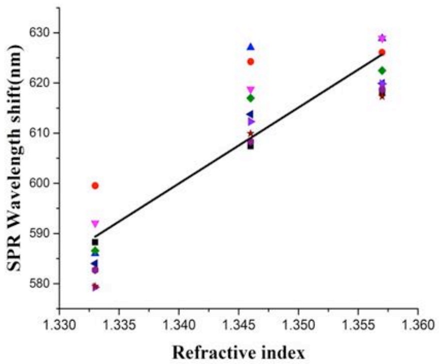


Fig. 5. (a) Transmittance of the reflection-based setup for different refractive indexes and (b) SPR wavelength shift vs refractive index for the reflection-based setup, for different repeated cycles (symbols) and linear fit (solid line).

In order to evaluate the behavior of the sensing fiber as a disposable probe, to be used in the reflection-based setup, repeated measurements have been carried out by dipping the fiber sensor into the solutions of Tab. 1 after it was disconnected/reconnected into the arrangement of Fig. 2. Fig. 7 depicts the wavelength shift for three cycles, showing that two repeated measurements out of three overlap well, whereas the third one is ~10 nm away, which is still quite high. The reflection-based setup demonstrates to be a convenient configuration for the interrogation of the SPR optical fiber sensor, since this can be easily connected/disconnected by movable connectors, but care shall be taken to avoid large misalignment that introduce large errors in the SPR wavelength reading.

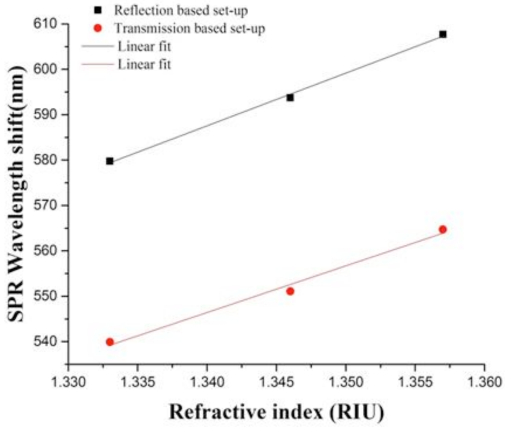


Fig. 6. SPR wavelength shift comparison between transmission and reflection based set-up.

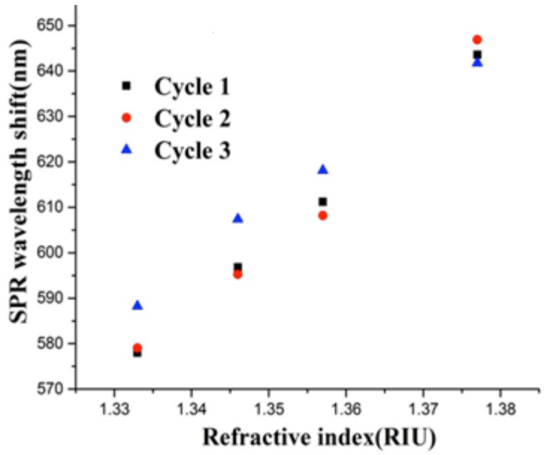


Fig. 7. Wavelength shift versus refractive index for repeated connections of the fiber probe in the reflection-based setup. The fiber is disconnected/reconnected at each cycle.

V. CONCLUSIONS

A comparison between the sensing characteristics of a surface plasmon resonance optical fiber sensor with transmission and reflection based setup has been presented. The sensors have been tested by measuring solutions with known refractive indexes from 1.330 to 1.377. The effect of misalignment of the source has been investigated for the transmission based setup, showing that it can heavily affect the measure. An improved sensitivity of the sensor in the reflection configuration has been assessed. The sensor has been tested as a disposable probe in the reflection based setup, showing that this arrangement is convenient, provided the connection into the measurement system is performed with good accuracy.

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