Forty-four pass fibre-optic loop for improving the sensitivity of surface plasmon resonance sensors

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Abstract
A 44-pass fibre-optic surface plasmon resonance sensor that enhances detection sensitivity according to the number of passes is demonstrated for the first time. The technique employs a fibre-optic recirculation loop that passes the detection spot 44 times, thus enhancing sensitivity by a factor of 44. Presently, the total number of passes is limited by the onset of lasing action of the recirculation loop. This technique offers a significant sensitivity improvement for various types of plasmon resonance sensors that may be used in chemical and biomolecule detections.

Keywords: surface plasmon resonance, fibre-optic surface plasmon sensors

1. Introduction
Surface plasmon resonance (SPR) sensors have been used for chemical and biological sensing for more than two decades [1–3]. Surface plasmons are charge density waves that propagate along the surface of metals, and are created when an optical wave at some incident resonance angle impinges a dielectric–metal–detection region structure [4, 5]. On resonance, the interaction of surface plasmon and the optical wave reduces the structure reflectivity. Simultaneously, the magnitude of the localized and exponentially decaying evanescent field on the detection region exhibits a maximum value at the gold–detection region interface. Perturbations of the evanescent field by the residing molecules shift the resonant angle [6].

SPR can be implemented using the grating coupled [4–7] or prism coupled [8] (Kretschmann) configuration. Here, we used Kretschmann’s configuration for demonstrating the 44-pass sensitivity. The standard Kretschmann configuration consists of a light source and a detector on opposite sides of a prism that allows for one reflection (one pass) of the optical beam off the gold layer. In an n-pass system, the light impinges on the detection region n times by multiple reflections. Recently, we reported on the performance of an SPR setup using a corner cube retroreflector replacing the photodetector to achieve four passes instead of one [9], and suggested an external fibre recirculating loop method to increase the number of passes beyond 4. In this paper, we demonstrate a working recirculating loop that achieves 44 passes, and we show the sensitivity scales with the number of passes. This technique detects both charged and uncharged analytes, in contrast to the field-assist technique reported in [10] that improves the sensitivity by attracting only charged analytes to the detection region.

A brief analysis shows the enhanced sensitivity for more passes. If the detected power \( P \sim R^n \), where \( R \) is the SPR reflectivity, then the fractional change in power, \( \delta P/P \), due to a resonant angle shift, \( \delta \theta \), is given by

\[
\frac{\delta P}{P} = n \left( \frac{d \ln(R)}{d \theta} \right) \delta \theta
\]

which increases with the number of passes \( n \).

2. Experimental setup
Figure 1 shows the SPR setup with the all-fibre recirculating loop. The SPR setup comprises a fibre-optic collimator on one side of the prism and a mirror reflector on the other side that reflects the beam back into the fibre collimator, resulting in a stand-alone two-pass configuration that has a lower optical back-coupling loss than the four-pass configuration of [9]. The
Properties of fibre components used here are reviewed in [11].

lower loss eases the burden on the optical amplifier used in this experiment.

The principle of the 44-pass operation is described below. Properties of fibre components used here are reviewed in [11]. Pulse generator 1 drives a laser diode (LD) to produce an optical pulse train with about 5% duty cycle. The pulse width is about 0.13 \( \mu s \). Pulse generator 2 is gated by this pulse train to produce a synchronized pulse train with a much longer pulse width \( T \) as shown, the purpose of which will be described later. The optical pulse incident on fibre coupler FC1 is split into two pulses. One pulse propagates towards port 1 of the optical circulator after traversing a polarization controller, PC3, and a fibre delay line. The optical pulse train proceeds towards the SPR setup by exiting port 2 of the optical circulator. The fibre collimator collimates the laser beam that impinges on the gold-coated BK-7 substrate. The beam reflected off the gold-coated substrate is reflected back to the fibre collimator by the mirror, retracing the original optical path. Thus, the SPR setup itself is a two-pass device. The optical pulse that is significantly reduced in amplitude due to SPR resonance effect and the back-coupling loss at the collimator re-enters the fibre loop via port 3 of the optical circulator. The pulse is amplified and restored to the initial amplitude by the erbium-doped fibre amplifier after passing through the electro-optic modulator (EOM). The pulse eventually reaches FC1 to complete one round trip. This returned pulse is again split into two pulses by FC1, one pulse is detected by a detector/amplifier module (PD), and the other pulse travels towards the SPR to repeat the process. Therefore, the detector/amplifier module detects a series of periodic pulses. The first pulse does not pass the SPR device. The second pulse passes the SPR twice, and the third pulse passes the SPR four times etc. The period is determined by the round-trip time through the fibre loop and the SPR setup. The added fibre delay line in the fibre loop is simply to assure that the 0.13 \( \mu s \) recirculating pulses are well separated in time.

The EOM functions as a loss-modulating optical switch. The switch is closed (low loss) when the gated electrical pulse applied to the RF port of the EOM is on, otherwise the switch is opened (high loss). The time duration of the gated pulse \( T \) determines the total number of passes of the SPR system. This switching action is important because the fibre loop with the optical amplifier comprises a fibre laser that can lase without any input, thus destroying the function of the SPR. The periodic opening of the EOM switch prevents lasing from occurring, but, as a compromise, limits the maximum number of achievable passes. Appropriate adjustment of the three polarization controllers, PC1, PC2 and PC3, ensures that the same optical polarization is maintained for every round trip of the recirculating pulse, and, also, the polarization is p-polarized at the SPR for exciting the surface plasmon [4].

3. Experimental results

The basic SPR function is first verified by measuring its one-pass characteristics by disconnecting the recirculation loop and by temporarily replacing the reflecting mirror on the SPR setup by a photodetector. The reflectivity versus incident angle profile is shown in figure 2 when DI water is dispensed onto the gold surface. One obtained the familiar SPR curve with a minimum reflectivity at resonance occurring at about 62.6° incident angle (corresponds to \( \theta \) in figure 2). Our diode laser wavelength is 1.53 \( \mu m \), compatible with erbium-doped fibre amplifier technology. At 1.53 \( \mu m \) wavelength, the resonance profile is sharper and the reflectivity dip is shallower than the response at the traditionally used wavelength of 0.78 \( \mu m \), which can be verified by simulation [9].

For multipass applications we rotate the collimator and the mirror to set the bias point at 0.17° (±0.02) below resonance, as indicated by the arrow in figure 2. The 44-pass response is first performed with DI water (18 MΩ cm quality) by dispensing it on the gold surface. The gain of the fibre amplifier is adjusted such that amplitudes of pulses for the case of water are approximately equal as shown by the lower trace in figure 3. Then the water is replaced with a 0.01% gram-salt (1.7 mM) salt solution, and its pulse train is measured. Both results are superimposed in figure 3. The presence of salt increases the solution’s index, and according to SPR theory, shifts the resonant angle to slightly larger angle, causing an increase in the reflectivity and optical signal when the set bias

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Figure 1. The multipass setup. Top: SPR, bottom: fibre loop, LD: laser diode, PD: photodiode and EOM: electrooptic modulator.

Figure 2. The one-pass reflectivity versus angle shift. Zero degrees corresponds to the resonance angle at 62.6°. The arrow represents the set bias angle at 0.17° below the resonant angle.
angle is below the resonance angle. The total number of pulses is 22 and the corresponding number of passes is 44, as each round trip of the pulse through the loop impinges on the gold surface twice. Figure 3 reveals the differential increase of the pulse amplitude with the number of passes for the salt solution over DI water. This result demonstrates the higher sensitivity for more passes.

It is noted that the increase in the magnitude of the base line with time, as observed in figure 3, is due to the temporal increase in amplified spontaneous emission from the fibre amplifier that ultimately will lead to self-lasing within the loop if the gated pulse width, $T$, is too long.

From figure 3, one deduces the fractional signal increase (dictated by equation (1)) of the salt solution with respect to DI water for the various pulses as $(P_{w,m} - P_{w,m})/P_{w,m}$, where $P_{w,m}$ and $P_{w,m}$ are measured peak amplitudes of the $m$th pulse for the salt solution and DI water respectively. The fractional increase, $(P_{w,m} - P_{w,m})/P_{w,m}$, is plotted in figure 4 as solid circles. Note that for two passes, the fractional signal increase is too small to be determined. But for 20 passes the fractional signal increase is about 0.55, and for 40 passes, the signal increase factor is 1.2, which is a factor of about 2 increase from 20 to 40 passes.

An SPR programme, which uses the SPR theory presented in the appendix, is used for predicting the fractional increase for the various passes. Quantities that need to be calculated are $[(R_{s})^{2m} - (R_{w})^{2m}]/(R_{w})^{2m}$, where $R_{s}$ and $R_{w}$ are one-pass reflectivities for the salt solution and water respectively. The $m$th pulse shown in figure 1 (not counting the first one) passes the SPR 2$m$ times, and the effective reflectivity is raised to $2m$ power. The program for calculating the reflectivity uses the transmission matrix method for plane waves. The original method can be found in [12]. Basically, the method assumes an incident and reflected electromagnetic field in any individual layer of a specified multilayer dielectric (including metallic) structure. By matching tangential components of $E$ and $H$ fields at the layers’ boundaries, one obtains an angle-dependent reflectivity. The details of the SPR calculation are given in the appendix.

Reflectivities, $R_{s}$ and $R_{w}$, are calculated for the ‘two structures’: BK7–Ti–Au–water and BK7–Ti–Au–salt solution. The water index $n_{w}$ is 1.3159, which is smaller at 1.5 $\mu$m than at visible wavelengths [14]. The salt solution index is taken as $n_{s} = 1.544$ is the index of salt at 1.5 $\mu$m, $n_{w}$ = 1.3159 is the index of water, and $x = 10^{-4}$ (salt weight fraction), the concentration of the salt solution. The predicted $\delta n$ is $2.3 \times 10^{-5}$, which agrees fairly well with the fitted value of $\delta n = 2.7 \times 10^{-5}$.

Other common parameters used in the calculation are the BK7 prism index: 1.500 65, the Ti complex index: $4.0 + i \times 3.8$ (thickness 10 nm) and the Au complex index: $0.2 + i \times 10.2$ (thickness 50 nm). These parameters at the 1.5 $\mu$m wavelength are obtained from [13].

4. Conclusion

In conclusion, a 44-pass all-fibre-optic technique for a surface plasmon resonance sensor that enhances detection sensitivity according to the number of passes is introduced for the first time. The technique employs a fibre-optic recirculation loop that passes the detection spot 44 times, thus enhancing sensitivity by a factor of 44. The key to the successful implementation of the concept is the gated switch that turns off the fibre loop to suppress lasing effects. This technique offers significant sensitivity improvements over traditionally used one-pass plasmon resonance sensors.

Presently, the total number of passes is limited by the onset of lasing action occurring after time $T$ ($T$ in figure 3). An obvious method to significantly increase the number of passes beyond what has been achieved here is to shorten the optical pulse to accommodate more pulses within the $T \approx 10 \mu$s time duration before amplified spontaneous emission becomes too serious. A corresponding increase in the detection bandwidth is also needed.

Appendix: SPR theory

The goal is to calculate the reflectivity of the multilayer structure depicted in figure 5, which shows the incident electric field $E_{i}$ and its propagation direction $k_{i}$ inside the BK-7 prism.
The thickness of the Ti-layer is 10 nm, and its dielectric constant is $\varepsilon_1 = n_1^2 = 4.0 + i \times 3.8$. The gold layer thickness is 50 nm and its dielectric constant $\varepsilon_3 = n_3^2 = 0.2 + i \times 10.2$. The water index is 1.3159. The salt solution index is 1.3159 + $\delta n$. $\delta n$ is varied to fit to the data.

Since it is well known that only p-polarized light can create a plasmon mode in the metal, we assume that the field is described by a plane wave with a p-polarized electric field in the $x$-$z$ plane of incidence.

With propagation constant $\beta$ describing a wave propagating in the $x$-direction, the electric field $E$ and magnetic field $H$, represented by $\psi(x, z)$ in any layer can be written in the form

$$\psi(x, z) = \Psi(z) e^{i \beta x}. \quad \text{(A.1)}$$

The scalar wave equation for $\Psi(z)$, according to Maxwell’s equations, gives

$$\frac{d^2 \Psi(z)}{dz^2} = (\varepsilon k_0^2 - \beta^2) \Psi(z), \quad \text{(A.2)}$$

where $\varepsilon$ is the dielectric constant of the individual layer, and $k_0 = 2\pi/\lambda$, where $\lambda$ is the wavelength. Referring to figure 5, the magnetic field $H$ is in the $y$-direction (perpendicular to the plane of incidence) because we assume a plane wave with a p-polarized electric field in the $x$-$z$ plane of incidence. Thus, $H_y(z)$ can be solved by equation (A.2), and the electric field $E_x$ and $E_z$ can be related to $H_y$ via Maxwell’s equation in any layer by

$$E_x = \frac{i}{\omega \varepsilon_0} \frac{\partial H_y}{\partial z}, \quad E_z = \frac{\beta}{\omega \varepsilon_0} H_y, \quad \text{(A.3)}$$

where $\omega$ is the angular frequency. Note that $H_y$, $E_x$, and $E_z$ are tangential components to the layers’ boundaries.

According to equation (A.2), $H_y(z)$ can be written as

$$H_y(z)_n = A_n \exp(i\beta_n z) + B_n \exp(-i\beta_n z), \quad \text{(A.4)}$$

with

$$\beta_n = \sqrt{k_0^2 \varepsilon_n^2 - \beta^2}. \quad \text{(A.5)}$$

Index $n$ labels the $n$th layer and $\varepsilon_n$ represents the dielectric constant of the $n$th layer, which is given by the square of the refractive index (or complex refractive index for the case of the metal layer). $A_n$ and $B_n$ refer to the forward and backward travelling wave respectively.

From (A.4) and (A.3), $E_x$ can be written as

$$E_x(z)_n = \frac{i}{\omega} \left( \frac{i \beta_n}{\varepsilon_n} \right) \left[ A_n \exp(i\beta_n z) - B_n \exp(-i\beta_n z) \right]. \quad \text{(A.6)}$$

Equations (A.4) and (A.6) are used to match the tangential components of the field $H_y$ and $E_x$ at boundaries between the $n$th and the $(n + 1)$th layers. One obtains

$$A_n \exp(i\beta_n z_n) + B_n \exp(-i\beta_n z_n) = A_{n+1} \exp(i\beta_{n+1} z_{n+1}) + B_{n+1} \exp(-i\beta_{n+1} z_{n+1}) \quad \text{(A.7)}$$

$$\frac{\beta_n}{\varepsilon_n} \{ A_n \exp(i\beta_n z_n) - B_n \exp(-i\beta_n z_n) \} = \frac{\beta_{n+1}}{\varepsilon_{n+1}} \{ A_{n+1} \exp(i\beta_{n+1} z_{n+1}) - B_{n+1} \exp(-i\beta_{n+1} z_{n+1}) \}, \quad \text{(A.8)}$$

where $z_n$ labels the coordinate of the $n$th and $(n + 1)$th layer boundary. Equations (A.7) and (A.8) can be written in a matrix form

$$\begin{pmatrix} A_n \\ B_n \end{pmatrix} = \begin{pmatrix} M_{n,n+1} & N_{n,n+1} \\ N_{n,n+1} & M_{n+1,n} \end{pmatrix} \begin{pmatrix} A_{n+1} \\ B_{n+1} \end{pmatrix}. \quad \text{(A.9)}$$

The elements in the $2 \times 2$ matrix $M_{n,n+1}$ can easily be identified from (A.7) and (A.8). Generally, if the $5$th layer is labelled as the gold layer, then the sample layer is the $S + 1$ layer. In our experimental case, $S = 2$, as shown in figure 5. From (A.9), one can write in a general form

$$\begin{pmatrix} A_0 \\ B_0 \end{pmatrix} = \begin{pmatrix} M_{0,1} & M_{0,2} & \ldots & M_{0,S+1} \\ N_{1,0} & N_{1,1} & \ldots & N_{1,S+1} \end{pmatrix} \begin{pmatrix} A_{S+1} \\ B_{S+1} \end{pmatrix} \equiv N \begin{pmatrix} A_{S+1} \\ B_{S+1} \end{pmatrix}. \quad \text{(A.10)}$$

Assume the sample layer is infinite in extent as is essentially the case in this experiment, then the backward propagating wave in the sample layer is zero, thus $B_{S+1} = 0$. The SPR field reflectivity $r = B_0/A_0$, noting that $E$ can be obtained from $H$ by equation (A.3). Thus, from (A.10)

$$r = \frac{B_0}{A_0} = \frac{N_{21}}{N_{11}}. \quad \text{(A.11)}$$

The SPR power reflectivity $R = |r|^2$ is then given by

$$R = \left| \frac{N_{21}}{N_{11}} \right|^2. \quad \text{(A.12)}$$

The matrix method is convenient because matrix multiplications can be called up directly from a calculation platform such as MathCad (the platform used in this calculation). Referring to the incident angle $\theta$ given in figure 5, the magnetic field $H_y(x, z)$ in the BK-7 prism layer can be written as

$$H_y(x, z) = \Psi(z) \exp[i\eta_0 k_0 \sin(\theta) x], \quad \text{(A.13)}$$

with

$$\Psi(z) = A_0 \exp[i\eta_0 k_0 \cos(\theta) z] + B_0 \exp[-i\eta_0 k_0 \cos(\theta) z]. \quad \text{(A.14)}$$

The quantity $\eta_0 k_0 \sin(\theta)$ in (A.13) is the propagation constant $\beta$ in equation (A.5). Here, $\eta_0$ is the refractive index of the BK-7 prism. With a MathCad program using formulae given above and the layers’ parameters given in the text, one calculates the reflectivity $R$ (and therefore $R^2m$) at the bias angle for values of the sample index that correspond to DI water or the salt solution as described in the text.
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