Surface plasmon resonance detection based on a phase method in the spatial domain

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ABSTRACT

A phase detection method is proposed and demonstrated to measure the response of surface plasmon resonance (SPR) in the spatial domain. In an experimental setup comprising a laser source, a launching optics, a gold coated SF10 glass plate, a coupling prism, a Wollaston prism and a CCD camera, a spatial interference fringes are recorded. Two such spatial interferograms, one including interference of p- and s-polarized waves from the SPR structure for air when the SPR phenomenon does not occur, and the other one for an analyte when the SPR phenomenon occurs, are used to detect the SPR phase shift at the source wavelength. The interferometry setup is used to measure the SPR phase shift for aqueous solutions of ethanol. The measurements are accompanied by theoretical modeling of the SPR responses using the material characteristics, that is, the refractive index of the SF10 glass, the complex refractive index of gold, and the refractive index of the analyte.

Keywords: surface plasmon resonance, phase detection, spatial domain, refractive index, water, ethanol

1. INTRODUCTION

Surface plasmon resonance (SPR) phenomenon has been already applied in many areas of sensing. It was used in measurements of concentration of ethanol,1 refractive index2 and also in biosensing.3 The phenomenon has also found its place in fibre sensing. The first publication about the SPR fibre sensor is from 19924 and from that time, many publications about SPR fibre sensing, including sensing of refractive index,5, 6 temperature6, 7 or concentration of glycerol,8 have been published.

The surface plasmons (SPs) can be described as oscillation of free electrons on the boundary that separates a dielectric material and a metal. Electromagnetic field of SPs has its maximum value on the boundary and exponentially decays into both media.9 The most widely used configuration for the surface plasmon excitation is the Kretschmann configuration which was introduced in 1968 by Kretschmann and Raether.10 It uses a prism made of glass with high refractive index on whose base is deposited a thin layer of metal. When a p-polarized light wave passes through the prism and totally reflects from the layer, an evanescent wave is generated which penetrates through the layer and excites the SPs on the other side boundary. When the tangential part of the wave vector of the incident light matches the wave vector of the SP wave,10, 11 the resonance condition is fulfilled and the SPR occurs, which manifests as a decrease in intensity of the reflected light and as a shift in its phase. The effect strongly depends on the local changes in refractive index of an analyte near the metal layer. One can detect resonance angle or resonance wavelength and also the phase shift both in spectral12 and spatial domains.

In this paper, a method of the SPR phase detection is introduced.13 It is based on spatial interference of two monochromatic plane waves when one of them is affected by SPR effect. This is manifested as a shift of the fringes in the interference pattern contrary the reference pattern where the SPR effect is not involved. The phases of both patterns are obtained by using a windowed Fourier transform and then the phase shift is determined for several types of analytes. In the first part, the theoretical modeling and image processing are introduced. In the second part, the experimental setup together with experiment procedure are described. Finally, in the last part, the results of experiment are presented.

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2. THEORY

In this part, a method of phase detection is introduced and accompanied with the theoretical modelling. The SPR structure under consideration is shown in Fig. 1. It consists of a substrate made of glass SF10, an adhesive layer of chromium of thickness 2 nm, an SPR layer of gold of thickness 44.8 nm, a thin layer of an effective medium with a thickness of 2 nm and an analyte as a superstrate. In the effective medium layer a surface roughness of the golden layer is involved.

![Fig. 1: The Kretschmann configuration and the structure used for modeling the SPR response in detail.](image-url)

2.1 Interference patterns

Suppose now that two monochromatic and linearly polarized plane waves, one of them is \( p \)-polarized and the other one is \( s \)-polarized, are reaching the thin layers in the structure on angle of incidence \( \phi \) which is very close to the resonance angle \( \phi_R \). Reflection from the layers is related to the complex reflection coefficients, \( r_p = \sqrt{R_p} \exp i \delta_p \) for \( p \) polarization, \( r_s = \sqrt{R_s} \exp i \delta_s \) for \( s \) polarization, where \( R_p, R_s \) are reflectances and \( \delta_p, \delta_s \) are phases of \( p \)-polarized and \( s \)-polarized light waves, respectively. The coefficients can be expressed by Fresnel formulas\(^\text{14}\) or Yeh matrix formalism\(^\text{14}\) with use of dispersion relations of the materials involved in the structure.\(^\text{15–17}\) The SPs are excited by the \( p \)-polarized wave and the phase shift \( \delta_{SPR} = \delta_p - \delta_s \) is introduced. By spatial splitting of the waves and taking projections of them in one direction of polarization and then combining them on a screen with angle \( \alpha \) between them, a spatial interference pattern can be obtained. The pattern is formed by fringes of equal thickness according to the interference law. The law limited only to one spatial dimension \( y \) which is perpendicular to the fringes can be expressed as

\[
R(y) = R_1 + R_2 + 2 \sqrt{R_1 R_2} \cos (\Delta \Phi(y) + \delta_{SPR}),
\]

where \( R_1 \) and \( R_1 \) are the reflectances of the interfering waves, \( \Delta \Phi(y) \) is the interference phase and \( \delta_{SPR} \) is the phase shift introduced by the SPR. The interference phase can be expressed as

\[
\Delta \Phi(y) = 2ky \sin \alpha,
\]

where \( \alpha \) is the half of the angle between the interfering beams and \( k \) is the wave number of the incident light. Two patterns are obtained, one for analyte, when the SPR does occur, and the reference one for air, when the SPR does not occur for the used wavelength and the angle of incidence \( \phi \). In Fig. 2 are two such model patterns, one for air and the other one for analyte (distilled water). The shift of the fringes is obvious in the second pattern contrary to the reference one, as well as decrease in the visibility.
Fig. 2: The reference interference pattern for air, \( \alpha = 7^\circ, \lambda = 637 \text{ nm} \).

Fig. 3: The interference pattern for analyte (distilled water), \( \alpha = 7^\circ, \lambda = 637 \text{ nm} \).

### 2.2 Image processing

In this method, a CCD camera is used for capturing raster images of the interference patterns and the images can be treated as RGB matrices. Since the used wavelength is \( \lambda = 637 \text{ nm} \), only the R- part of the matrix is relevant. Each row of that part represents the intensity as a function of the pixel number (interferogram). In Fig. 4 is the example of the interferogram obtained from the model fringe pattern shown in Fig. 3. The red line in the picture is the background intensity and it needs to be removed to obtain an interference signal, which can be then processed by a windowed Fourier transform (WFT).\(^{18}\)

![Interferogram](image)

Fig. 4: Intensity as a function of the pixel number. The red line is the background intensity.

The intensity dependent on the pixel number \( n \) can be expressed as

\[
I(n) = a + b \cos \beta(n),
\]

where \( a \) is the background intensity, \( b(n) \) is a constant term related to the visibility of the fringes and \( \beta \) is the overall phase. The term \( a \) can be obtained using the Fourier transform and the interference signal can be then determined as

![Unwrapped phase](image)

Fig. 5: Unwrapped phase obtained by the WFT.
The phase from the interference signal can be obtained by using the WFT, and an example of retrieved phase from the interferogram shown in Fig. 4 is depicted in Fig. 5. In Fig. 6 is the reconstructed interferometric signal with the retrieved phase together with the original one.

\[ S(n) = \frac{I(n)}{a} - 1. \]  \hspace{1cm} (4)

From the retrieved phases for both interference patterns, the phase shift \( \delta_{SPR} \) is determined (Fig. 7). At the edges, a significant increase and decrease in the phase shift can be seen, which is due to finiteness of the processed signal.

### 3. EXPERIMENT

In this section, the experimental set-up and the measuring procedure are introduced. The set-up is seen in Fig. 8. It consists of light source \( LS \) (laser diode DL-4039-011, Sanyo), polarizer \( P \) and analyser \( A \) (LPVIS050, Thorlabs), Wollaston prism \( WP \) (WP10, Thorlabs), a prism with an SPR structure, a CCD camera (PL-B952U, Pixelink), a computer and lens \( L \). To avoid the negative external disturbances, the set-up is on an optical table.

In the first step, the interference pattern for air is recorded in the following procedure. A light beam from the light source (\( \lambda = 637 \text{ nm} \)) passes through the polarizer with polarization axis oriented 45° with respect to the plane of incidence, so both polarizations \( s \) and \( p \) are generated. Then the light beam is refracted on the air/prism boundary and reaches the SPR structure at angle of incidence \( \phi \). After reflection from the structure, the beam refracts on the prism/air boundary and is splitted into two beams by the Wollaston prism. The beams then pass through the analyser with polarization axis also oriented 45° with respect to the plane of incidence, so the components of polarization are projected to direction of the analyser axis. Using the lens, the beams are combined on the CCD camera where they interfere. The interference pattern serves as the reference pattern, because the SPR does not occur for the used wavelength and angle of incidence.

In the second step, the analyte is led directly on the gold layer in the sensing chamber and the pattern for analyte is recorded with using the same procedure as in the first step. As the analytes, solutions of distilled water and ethanol are used. The weight concentrations of ethanol are 2.5, 5, 10, 20, 30, 40 and 50 wt\%. Both patterns are then processed by the WFT and the phases \( \beta \) and \( \beta_R \) are obtained. Finally, the phase shift \( \delta_{SPR} = \beta - \beta_R = \delta_p - \delta_s \) is determined.
4. RESULTS AND DISCUSSION

Reference interference pattern for air is shown in Fig. 9 and similarly, the one for analyte with 50 wt% of ethanol is shown in the Fig. 10. For the analyte, decrease in the visibility of the fringes in the pattern is evident as well as their shift. It can be also seen that diffraction artefacts are involved that are attributed to some impurities on the optical elements.

Finally, in Fig. 11 are the phase shifts for seven analytes with different weight concentrations of ethanol. The range of pixel number is only from 400 to 600, since the errors are increased towards the edges. Average values of the phase shifts as a function of ethanol concentration are shown in Fig. 12. As is seen in the figure, non-linearly dependence starts from 30 wt% and it is limitation of the method.
5. CONCLUSIONS

In this paper, a method of SPR phase detection with using spatial domain interferometry has been presented. In the experimental set-up, a spatial interference patterns have been recorded. Two such spatial interferograms, one including interference of $p$- and $s$-polarized waves for air, when the SPR phenomenon does not occur, and the other one for an analyte when the SPR phenomenon occurs, have been used to detect the SPR phase shift at the source wavelength. As analytes, solutions of distilled water and ethanol with weight concentrations 2.5, 5, 10, 20, 30, 40 and 50 wt% have been used. The obtained phases have been disturbed with a significant noise so that a new method of filtering the interference patterns needs to be applied. Since the dependence of the phase shift on the concentration is linear in a narrow interval, the method is suitable only for a limited range of concentrations. Results are important from the point of view of new sensor developments and designs.

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