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Designing a curved surface SPR device

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Abstract

A spherically curved surface plasmon resonance (SPR) surface is proposed theoretically and tested experimentally. The format achieves multi-angle SPR, where the angular modulation of the incident beam is performed by the geometrical shape of the SPR surface, not external to the sensor optics, thus achieving a robust compact design. The position of the imaged SPR band reflected from the surface is shown to be sensitive to adsorption of low-density lipoprotein (LDL) and exposure to nitrogen dioxide. Proposals for prototype sensors heads based on this concept are also shown.

Keywords: SPR; Multi-angle; Curved interface

1. Introduction

Sensor devices using evanescent fields for surface plasmon resonance (SPR) and SPR-like detection, can be distinguished according to the method used to couple light from a source into the optical 'transducer'. A relief grating to couple the light has been used to view resonance peaks in thin waveguides [1], and also to excite SPR on thin gold films [2,3], whereas prism coupling has been employed for the two most established techniques, used for SPR. In the Kretschmann–Raether geometry, the thin gold film is in direct optical contact with the prism (or other coupling device) [4,5], and for Otto's configuration there is an air gap between the two [6,7]. In the development of these types of optical sensors it is the Kretschmann–Raether geometry that has been most popular and most widely adapted.

One driving factor in considering modifications or other designs is the possibility of obtaining more information from analysis using SPR as the measurement technique while minimising the size and complexity of the device. Typically for SPR a way to increase the amount of information extracted from the system is to vary the incident angle of the light or its wavelength, since both affect the intensity of the reflected light. A commonly used method to affect a multi-angle response is to optically modify the beam of incident light, which is then coupled by the prism onto the gold surface, and record the information from the surface on a linear detector array [8–10]. This technique is used by most of the commercially available SPR devices [11,12]. Multi-wavelength SPR has also been achieved with white light sources (halogen and white light LEDs), which emit over a large section of the visible spectrum, and are able to excite surface plasmons. In these systems reflected intensity at a given wavelength is found using a photodetector behind a monochromator or by using a spectrograph [13–15].

These methods achieve multi-angle or multiwavelength measurement without rotation of the prism assembly with the design requiring consideration of the geometry of the optical components to achieve success. This concept has also been included in the miniaturisation of SPR devices by the use of waveguides [16], including light pipes [17,18] and optic fibres [19,20]. In this work we have examined whether a robust optical geometry can be achieved whereby multi-angle measurement can be generated from a single incident angle, by the internal inherent optical properties of a simple device design.

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2. Materials and methods

All gold films were prepared using 99.9994% Gold (Alfa Aesar) in an Edwards Auto 306 vacuum evaporator, the gold thickness as measured during evaporation on a FTM7 crystal monitor. The slides and the lens were coupled to the prisms using Benzyl Alcohol ($n = 1.540$, Fluka 99%). All planar SPR measurements were taken on a conventional optical bench. The details have been reported elsewhere [21]. Briefly it uses a 4 mW He–Ne laser incident through polarisation optics onto a prism-flow-cell unit mounted on a computer-controlled rotating stage. Detection in this system is by a photodiode which outputs to a computer controlled lock-in amplifier, the data being stored as a comma delimited ASCII text file.

The experimental data from reflection at a plano-convex lens was obtained using the Portable Attenuated Total Reflection Optical Bench (PATROB) built by Alphasense, Dunmow, Essex, UK. This has the same configuration as the conventional ATR optical bench described previously [21], with the following changes: the angle of the source and detector was varied in tandem under a manual micrometer control mechanism. Their tangential and radial locations can be adjusted by a few mm to ensure that a signal is seen. The light source was a 635 nm, 1 mW, polarised laser (Coherent). For the purpose of this work, the prism/flow cell assembly for standard SPR measurements on planar Au slides was modified to allow a prism coupled to a plano-convex lens. For measurements in air a 45° prism was used ($n_D = 1.517$, BK7), this is because the SPR minima occurs at ∼43–44° the apex angle of this prism is therefore chosen to minimise the refraction at the initial air/prism interface when light is incident at these angles. Detection was performed by a black and white CCD camera (Phillips VCM36), mounted on its side, so that all images shown from the PATROB are rotated by 90°. The camera was subject to a +5 V bias voltage to remove the effects of its in-built auto-gain. This camera emitted a PAL video signal which was captured using in-house software from an image grabber card (Aspro Technologies). The captured images were in a windows bitmap format and could be loaded into and analysed in Matlab (The Mathworks).

The plano-convex lens was obtained from Comar instruments, Cambridge UK, based on specifications generated by computer modelling. The lens ($n_D = 1.517$, BK7) had a diameter of 16 mm, a centre thickness of 2.5 mm and an edge thickness of 1.5 mm, this gives a radius of curvature $R = 32.5$ mm. The plano-convex lens was coated with a thin film of gold and coupled to the prism in the PATROB flow cell.

A 'solid-block' integrated attenuated total reflection (ATR) design was based on a plano-convex lens (Comar) of the same size as above. The other specifications and dimensions for the device were derived from computer modelling of the optical device using a custom Ray-Tracing algorithm in Matlab as described later. A plano-concave lens of diameter 10 mm, centre thickness 1.5 mm, and edge thickness 3.2 mm was used and the central glass block was formed from a cuboid with dimension 16 mm × 16 mm × 9.3 mm, with facets ground to angles of 45° along two of the edges. The curved sampling surface of this device was coated with a 40 nm gold film to achieve SPR and was interrogated with a 632 nm He–Ne laser beam. The detection of the light was achieved using a CMOS chip (17 mm × 17 mm area) from a small camera (Misumi MO-S617G), which emits a PAL signal. Images from this camera were captured using a video card (Pinnacle, PCTV) and were analysed in Matlab.

For testing nitrogen dioxide exposure, nitrogen (zero grade, BOC) and nitrogen dioxide (1 ppm in N_2, BOC) were passed through the PATROB flow cell at the same flow-rate; exposure times were all 10 min. An image of the reflected spot was taken every 10 s for 50 min. From the recorded images SPR curves were constructed: the location of the SPR minimum was followed by fitting the curves with a Fresnel-like equation [22], these values were then converted to the approximate angle shift using the factors described above.

The LDL solution used was derived from human blood samples and isolated as described previously [23]. The LDL adsorption on the gold film was carried out from a 0.1 mg/ml solution of LDL under stop flow conditions. The flow cell was filled with the LDL solution and the flow stopped for 5 min, before the cell was rinsed out with UHP water. The flow cell was then dried using a nitrogen flow for 10 min. Adsorption of LDL to a planar gold film was confirmed using the conventional optical bench. The reflection profile for the 'blank' gold slide was recorded in air before and after exposure to LDL solution.

3. Theory

Parallel light incident on a spherically curved surface is focused to a point [24], due to the changing surface normal across the curve. Thus, if the light source is placed off axis, hitting at an oblique angle, different parts of the beam hit the surface with different incident angles. This is demonstrated in Fig. 1a where two rays of a beam are incident, at an angle of θ to the y-axis, on the curved surface. The ray incident at the origin is locally incident at θ, but the ray at a diameter w in the beam is incident an angle γ (Fig. 1a) to the local surface normal of the curve. This angle is dependent on the changing value of α, which can be derived from x', the 'horizontal' distance along the x-axis at which the ray hits (Eq. (1)), where R is the radius of curvature.

$$\gamma = \theta - \alpha = \theta - \sin^{-1}\left(\frac{x'}{R}\right) \tag{1}$$

Variation in incident angle, γ, across the beam width, w, can be related to x' by finding the intersection between the equation of the curved surface (Eq. (2)) and the straight line of the ray (Eq. (3)).

$$x^2 + (y - R)^2 = R^2 \Rightarrow x^2 + y^2 - 2yR = 0 \tag{2}$$