

Demonstration of low-cost and compact SPR optical transducer through edge light coupling

Manjunath Somarapalli¹ ✉, Kartik Koul², Rimli Lahon³, Sakoolkan Boonruang⁴, Waleed S. Mohammed¹

¹BU-CROCCS, School of Engineering, Bangkok University, Phatumthani 12120, Thailand

²Centre for Nanotechnology Research (CNR), Vellore Institute of Technology, Vellore 632014, India

³Amity Institute of Nanotechnology, Amity University, Uttar Pradesh, Noida 201303, India

⁴Photonics Technology Laboratory, NECTEC, Pathumthani 12120, Thailand

✉ E-mail: s.manjunath005@gmail.com

Published in Micro & Nano Letters; Received on 2nd February 2017; Revised on 31st March 2017; Accepted on 4th April 2017

This work demonstrates experimentally a simple and practical low-cost surface plasmon resonance (SPR) system towards sensing applications. The cost is minimised through the elimination of many bulky optical components such as collimation lenses and prism. An off-shelf white light-emitting diode is directly coupled to a gold coated glass slide at a proper incident angle, while the output is collected with a bare multimode optical fibre mounted at the same incident angle and connected to a commercial spectrometer. Proper alignment of the components is achieved through building a mount using three-dimensional printing technology. The developed system is tested with glycerol solutions having different concentrations and the detection limit measured in the order of 10^{-4} RIU (refractive index unit). The system cost can be further reduced through mass production and when a low-cost in-house spectrometer is built while keeping the same precision.

1. Introduction: Surface plasmon resonance (SPR) is one of the most commonly used optical transducer in various sensing applications [1, 2]. Its usage has been demonstrated for wide range of applications such as detection of heavy metal ion in liquid [1], detection of harmful gas in the environment [2], and biosensor in medical purpose (hepatitis B for instance) [3] as well as food pathogen [4]. SPR system senses the changes on the surface of the device. Changes in the surrounding refractive index alter the resonance condition; the change of the system response can be examined through monitoring the intensity modulation [5], angular modulation [6], wavelength modulation [7], or phase and polarisation modulation [8]. In most of these monitoring schemes, large incident angle is required for SPR excitation. This is typically achieved using a prism [2]. This requires precise mechanical mounting to place the light source, prism, and the detector at proper angles. Using replaceable SPR chips commonly require applying an additional layer of index matching gel in between. One approach to eliminate the need of bulk prism is through placing diffractive optical coupling element on the back of the SPR chip itself [9]. This serves for light coupling and signal collection. In this approach, a diffraction grating is generated in a photo-resist and then a layer polymer is added on top. A thin metallic film is coated on the polymer. Another approach is by using waveguides for optical coupling. Using multimode fibre, the core region is exposed and coated with metal to allow for surface plasmon excitation from the guided modes [10]. In all of these approaches, the focus was on the system compactness. In terms of cost reduction, there has been several research works that proposed different inexpensive schemes. A free-space spectrum based SPR system was developed by Rampazzi *et al.* [11]. It consists of a pair of prisms, beam splitter, and a set of lenses for light coupling and signal collection. It was demonstrated to achieve sensitivity in the order of 10^{-5} RIU (refractive index unit) keeping the cost, without the external spectrometer, in the range of 600\$. Another low-cost and prism-based SPR system, referred to as SPREETA, was demonstrated by Texas Instruments [12]. In the system, light source, polariser, and an array of detectors are moulded in a plastic prism. An SPR chip is placed on the surface of the prism

and the achieved sensitivity is in the order of 10^{-7} . Since all the components are moulded to the plastic prism, the fabrication process of such systems is expensive. The cost is however kept cheap for the mass produced device. In waveguide-based compact systems, multimode optical fibre waveguides [13] are used. In the fabrication process, cladding layer is removed and a layer of polymer is coated between the fibre core surface and metal layer. An additional receptor layer is coated on top of metal for a specific bio-chemical application. The light coupling and reception is controlled by using Arduino.

This Letter demonstrates an alternative approach with low cost for both fabrication technology and system. In the proposed system, a gold coated glass substrate is used as slab waveguide. The guided light in the glass slide is used to excite the SPR signal. This configuration minimises the number of required components. The need of external coupling elements (such as prisms, lenses, or diffractive optical elements) is eliminated though exciting from the edge of the glass slide as shown in Fig. 1. The output is collected with a bare large core fibre placing it at proper angle in close proximity to the glass slide. To achieve proper system alignment, a mounting system was built as a one piece utilising three-dimensional (3D) printing technology which is easy and low-cost approach. Using the proposed scheme, the cost of the system without the external spectrometer is reduced to <30\$, which can be further reduced by mass production. The developed system performance is tested with different concentrations of glycerol solutions to observe the sensitivity.

2. Concept and fabrication: In typical SPR system, a prism is used as a coupling device to ensure light incidence at large angles on the gold surface. The use of index matching gel between a replaceable SPR chip and the prism requirement adds few steps to the measurement scheme. It as well requires a cleaning process. The use of prism is due to the fact that working angles are larger than the total internal reflection between substrate and surrounding. One way to eliminate the need of coupling components is through excitation from the side of the SPR chip itself. As shown in Fig. 1, at proper incident angle, light refracting at the gold interface will be at the desired SPR angle.

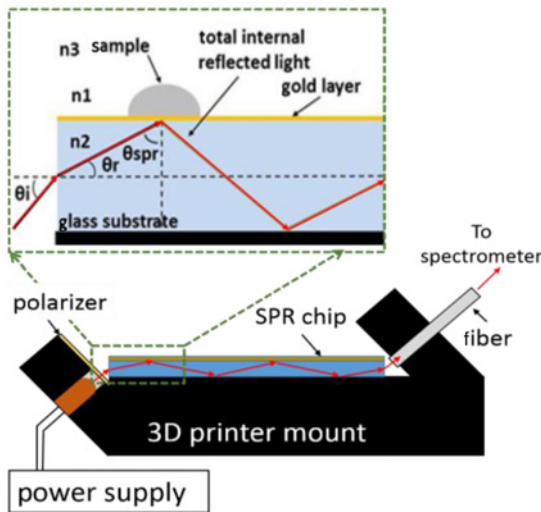


Fig. 1 Concept of refraction and total internal reflection of light in a glass substrate, supporting SPR phenomenon to occur

In Fig. 1, a transverse-magnetic (TM) polarised light is incident at an angle θ_i . The light refracts at an angle θ_r inside the glass substrate causing an angle θ_{spr} at the gold substrate. θ_i is designed to ensure that θ_{spr} excites SPR in the red range (660 nm). Using Snells' law

$$\theta_i = \sin^{-1} \left(\left(\frac{n_2}{n_1} \right) \sin(\theta_r) \right) \quad (1)$$

where n_1 is refractive index of air, and n_2 is refractive index of glass slide. The SPR angle with respective refractive angle is given by

$$\theta_{spr} = 90 - \theta_r \quad (2)$$

Incidence from air ($n_1 = 1$), (1) becomes

$$\theta_i = \sin^{-1} \left(n_2 \cos(\theta_{spr}) \right) \quad (3)$$

In (3), θ_{spr} is calculated using T-matrix approach [9] for a structure composed of a gold film between two dielectric media: glass substrate and air cladding region. The internally reflected light comes out of waveguide at an angle, θ_i . This light is collected directly by a fibre probe for spectral analysis. Upon change in refractive index from n_2 to n_3 , the SPR condition is changed and can be observed from the light collected. The shift in SPR condition for change in refractive index helps us in determining the sensitivity of the system. Following this approach, the required components can be reduced to gold coated slab waveguide, light-emitting diode (LED), polariser and fibre probe, to build SPR sensing system; therefore reducing the cost of the system. The fabrication of SPR chip is performed using DC sputtering. Process involves cleaning of glass substrate and coating a thin layer of chromium (~ 5 nm) followed by deposition of gold layer to a thickness of ~ 50 nm. Chromium is used to provide adhesion of gold to the silica glass substrate. The fabricated SPR chip is then mounted on 3D printed (using XYZ DaVinci Jr 1.0) structure as shown in Fig. 2.

An off-shift 50 mA white light LED is used as a source and a linear polariser sheet is placed in front of it to ensure TM excitation. The system is designed to provide an SPR dip at 660 nm wavelength of light when water ($n = 1.33$) is placed on the gold surface. Therefore, the angles are computed to be $\theta_{spr} = 73^\circ$, $\theta_r = 17^\circ$, and $\theta_i = 26^\circ$. Designing of the structure is done considering the computed incident angle for θ_i .

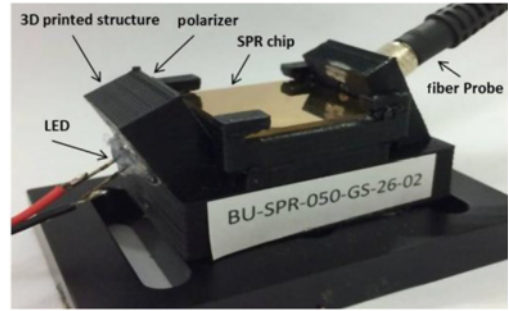


Fig. 2 Realised SPR system using 3D printed structure and assembly of components to perform the experiment

In the printed structure, the distance between the polariser and the slab waveguide is kept as close as possible to avoid dispersion of light and to ensure higher coupling into the substrate. The slab waveguide is placed on the printed structure at a computed height, so that light couples from the centre of the waveguide. The light propagating inside the slab waveguide undergoes total internal reflection and comes out at an angle equals to θ_i . At the receiver section, a small hole with a diameter that equals the fibre probe is placed in the 3D printed structure. The hole is designed such that the fibre probe placed at an angle equals to θ_i and it is kept as close as possible to the waveguide to maximise the light collection. The produced prototype has a dimension of $5 \times 3 \times 3$ cm.

It is worth mentioning that in this low-cost prototype, minimal optical components are used. Prisms and coupling lenses are eliminated through the proper angle alignment for the coupling and signal collection. This system is tested with solutions of different refractive indices to determine the measurement sensitivity.

3. Experiment and results: The theoretical analysis is done by simulating the reflection spectrum from the gold film when the incident angle equals to $\theta_{spr} = 73^\circ$, considering the thickness of the gold surface to be 50 nm with a refractive index of the glass substrate to be 1.3 mm and also the refractive index of dielectric interface with gold to be 1. In Fig. 3 (blue line), the obvious dip is observed when water is used.

The experimental results show that the SPR wavelength occurs around 600 nm. The mismatched resonance wavelength might be due to different excitation angle. The change in angle may be due to resolution of 3D printer that affects the position of light source and also may be due to change in θ_i due to positioning of the SPR chip in comparison to the light source. To match the experimental result, theoretical analysis indicates that the incident angle is 76° . Using (3), this corresponds to $\theta_i = 21.3^\circ$ ($\sim 5^\circ$ error).

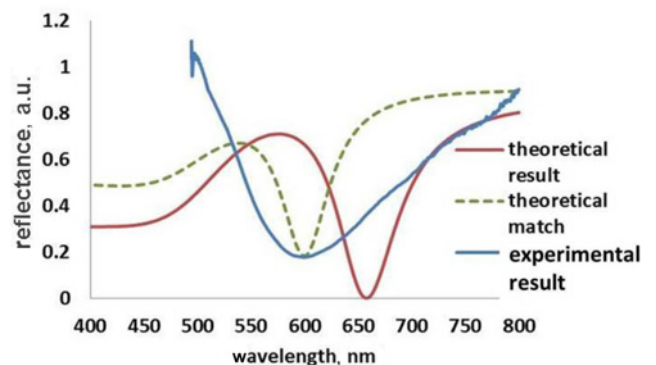


Fig. 3 Spectrum obtained from theoretical and experimental results

The experimental result shows as well a broader dip. Since a normal LED is used as light source, the light propagating inside the slab waveguide diverges with finite angular spectrum. The total response is then a convolution of the reflectance spectra of individual angular components. This diverging light results in broader output spectrum. This broadening however does not affect the dip wavelength (around the mean direction of propagation).

The developed system was tested with different solutions to estimate the sensitivity: water with refractive index (n) of 1.33244, glycerol solution with 5% concentration in deionised water with $n = 1.33779$, 10% with $n = 1.34788$, 15% with $n = 1.35127$, and 20% with $n = 1.35715$.

With air as reference, a drop of water is placed on the gold surface. The resultant spectrum is collected 15 times at a time interval of 1 min for each reading. The water is then replaced with glycerol solution with 5% concentration and spectrum is collected as before. The glycerol solution is then replaced by water and the output spectrum is recorded. This process is repeated for all the concentrations of glycerol solutions. The spectral data of water is collected every time after recording spectral response of glycerol, to check the system stability. The plot of dip SPR wavelengths obtained from recorded spectral response are shown in Fig. 4. The inset in the figure shows the recorded spectra for different concentrations.

In Fig. 4, w represents water; G5, G10, G15, and G20 represent concentration of glycerol as 5, 10, 15, and 20%, respectively. The refractive indices of these solutions are: 1.33244, 1.33779, 1.34788, 1.35127, and 1.35715, respectively. The plot for average of peak wavelengths with respect to refractive index is shown in Fig. 5. The results indicate that glycerol solution with higher concentration shows high standard deviation compared with lower concentration (standard deviation of 20% is almost five times

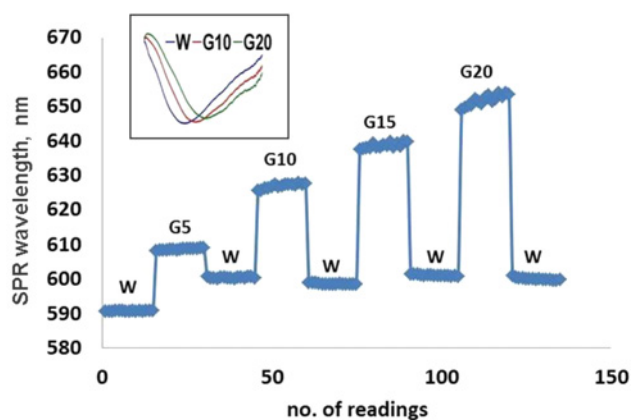


Fig. 4 Plot showing peak wavelengths recorded for different solutions

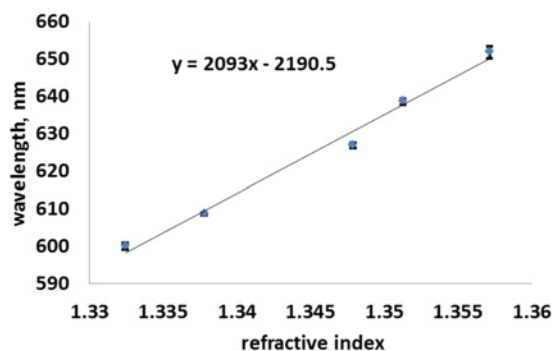


Fig. 5 Plot of average of peak wavelengths of different solutions with respect to their refractive index

larger than 5% and twice of 15%). Using a linear fitting of the results, the limit of detection (RIU) of the system is found to be 7.28×10^{-4} RIU.

In our system, though the result is one order of magnitude less than the typically reported system, it is still can be effectively used in field application where threshold detection is needed. Chemicals such as fluorine, zinc, and so on, present in underground water or water from wells are high enough to kill life upon exposure. The average intake of fluorine should be 3 mg for females and 4 mg for males [14], whereas according to World Health Organization (WHO) report published in 2006, water samples from various developing countries showed an average of 40 mg/l. In case of zinc, average intake should be 8 mg for females and 11 mg for males [14]. According to report published by WHO in 2003, Finnish survey of over 6000 wells had zinc content of 24 mg/l. Lower concentration of chemicals such as mercury vary from 1 nM to 1 mM in polluted water. Change in 100 μ M of mercury results in change of refractive index of order 10^{-4} . The sensor system with sensitivity in the order of 10^{-4} can be used for detection of chemicals whose presence is as low as 100 μ M [15, 16]. Quick results of yes, no, or not certain are enough. Towards the detection of very low concentrated pollutants, the polymer layer thickness on top of gold layer has to be modified. With 100% certainty of yes and no answers, sending samples for lab check can be minimised. The certainty of detection can be guaranteed due to repeatability and consistency of the proposed system as the mounting is made of one piece. The low cost allow the reading head to be replaceable after short-term use. This can as well help minimising the cleaning process due to surface contamination. It is as well worth mentioning that all the experiments were performed in ambient conditions. With an acceptable level of sensitivity, the system was proved to be robust and suitable for outdoors and portable applications.

Reducing the RIU unit to the order of 10^{-5} or even higher can be achieved with further optimisation of the gold layer thickness (for best spectral response), introducing microfluidic channel, and covering the system from ambient conditions. By increasing the film thickness up to around 65 nm, the sensitivity of the system can be increased higher order of RIU [17]. Introducing microfluidic channel for sample flow on top of gold layer limits area of interaction between gold layer and sample to reduce the effect of angular divergence; therefore further more improving the sensitivity of the system. Since the experiment is performed in ambient conditions, the humidity, atmospheric temperature, and pressure, affects the sensitivity of the system. Therefore by eliminating these parameters by providing a complete package to the system, the system sensitivity and precision can be further improved.

Finally, in terms of cost, the proposed system (referred to in the table as BU-SPR) is compared with two developed low-cost systems in Table 1.

The cost of SPREETA is considered to be ~ 50 \\$, only if the system is mass produced, whereas the lab prototype is estimated to be < 600 \\$, due to expensive components. With similar factor, our system (BU-SPR) cost can be reduced to < 10 USD with mass production.

4. Conclusions: The demonstration of low-cost, robust, and portable SPR system was realised. The cost was mainly reduced by using gold coated glass slide using DC sputtering. Minimum number of components was used and the mounting structure was designed and 3D printed to provide the required mechanical alignment and support. Off-shell LED was used with no prism, index matching gel, and collimators were used for coupling. The refraction from the glass slide tip was used to incident light on to the gold surface at a particular angle. The spectrometer probe was positioned close to the edge of the glass slide to collect the SPR signal. The developed system was tested with glycerol solutions of concentration 5, 10, 15, 20% and with water. The developed

Table 1 Specifications of different SPR systems

	Components	RIU	Cost, \$	Dimensions, cm ($W \times L \times H$)
system by Sara Rampazzi [11]	SPR chip, beam splitter, prism, index matching gel and lens	6×10^{-5}	< 600	$17 \times 15 \times 6$
SPREETA 2000 [12]	SPR chip, prism, photo-diode array, index matching gel and mirror	1.8×10^{-7}	~50	$4.2 \times 1.5 \times 3$
BU-SPR	SPR chip	7×10^{-4}	< 30	$5 \times 3 \times 3$

system showed better stability and good sensing with RIU in the order of 10^{-4} . Upon mass production, cost of the developed system can be further reduced, and the sharpness of the SPR dip can be increased by increasing the thickness of the gold layer.

5. Acknowledgments: The authors thank Ms Nantarat Srisuai, King Mongkut's University of Technology, Thonburi, Thailand and National Electronics and Computer Technology Center (NECTEC), Thailand.

6 References

- [1] Kyaw H.H., Boonruang S., Mohammed W.S., *ET AL.*: 'Design of electric-field assisted surface plasmon resonance system for the detection of heavy metal ions in water', *AIP Adv.*, 2015, **5**, p. 107226
- [2] Manera M.G., Rella R.: 'Improved gas sensing performances in SPR sensors by transducers activation', *Sens. Actuators B, Chem.*, 2013, **179**, p. 31
- [3] Choi Y.-H., Lee G.-Y., Ko H., *ET AL.*: 'Development of SPR biosensor for the detection of human hepatitis B virus using plasma-treated parylene-N film', *Biosens. Bioelectron.*, 2014, **56**, 12485–12492
- [4] Wang Y., Knoll W., Dostalek J.: 'Bacterial pathogen surface plasmon resonance biosensor advanced by long range surface plasmons and magnetic nanoparticle assays', *Am. Chem. Soc.*, 2012, **84**, (19), pp. 8345–8350
- [5] Nizamov S., Scherbahn V., Mirsky V.M.: 'Self-referencing SPR-sensor based on integral measurements of light intensity reflected by arbitrarily distributed sensing and referencing spots', *Sens. Actuators B*, 2015, **207**, pp. 740–747
- [6] Liedberg B., Lundstrom I., Stenberg E.: 'Principles of biosensing with an extended coupling matrix and surface plasmon resonance', *Sens. Actuators B, Chem.*, 1993, **11**, pp. 63–72
- [7] Zhang H., Song D., Gao S., *ET AL.*: 'Enhanced wavelength modulation SPR biosensor based on gold nanorods for immunoglobulin detection', *Talanta*, 2013, **115**, pp. 857–862
- [8] Nelson S.G., Johnston K.S., Yee S.S.: 'High sensitivity surface plasmon resonance sensor based on phase detection', *Sens. Actuators B, Chem.*, 1996, **35–36**, pp. 187–191
- [9] Thirstrup C., Zong W., Borre M., *ET AL.*: 'Diffraction optical coupling element for surface plasmon resonance sensors', *Sens. Actuators B*, 2004, **100**, pp. 298–308
- [10] Hu T., Zhao Y.: 'An-ning Song: Fiber optic SPR sensor for refractive index and temperature measurement based on MMF-FBG-MMF structure', *Sens. Actuators B, Chem.*, 2016, **237**, pp. 521–525
- [11] Rampazzi S., Danese G., Leporati F., *ET AL.*: 'A localized surface plasmon resonance-based portable instrument for quick on-site biomolecular detection', *IEEE Trans. Instrum. Meas.*, 2016, **65**, (2), pp. 317–327
- [12] Chinowsky T.M., Quinn J.G., Bartholomew D.U., *ET AL.*: 'Performance of the Spreeta 2000 integrated surface plasmon resonance affinity sensor', *Sens. Actuators B*, 2003, **91**, pp. 266–274
- [13] Cennamo N., Massarotti D., Monica A.D., *ET AL.*: 'A simple Arduino-based configuration for SPR sensors in plastic optical fibers'. *Fotonica AEIT Italian Conf. on Photonics Technologies*, 2015, Turin, 2015
- [14] Goldman L., Schafer A.: 'Goldmans cecil medicine', Chapter 225, eBook ISBN: 9780323295253, 25 July 2011
- [15] Chah S., Yi J., Zare R.N.: 'Surface plasmon resonance analysis of aqueous mercuric ions', *Sens. Actuators B*, 2004, **99**, pp. 216–222
- [16] Zhang P., Chen Y.-P., Wang W., *ET AL.*: 'Surface plasmon resonance for water pollutant detection and water process analysis', *Trends Anal. Chem.*, 2016, **85**, pp. 153–165, <http://dx.doi.org/10.1016/j.trac.2016.09.003>
- [17] Suzuki H., Sugimoto M., Matsui Y., *ET AL.*: 'Effects of gold film thickness on spectrum profile and sensitivity of a multimode-optical-fiber SPR sensor', *Sens. Actuators B*, 2008, **132**, pp. 26–33