High sensitivity surface plasmon resonance (SPR) refractive index sensor in 1.5 μm

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ABSTRACT

A surface plasmon resonance (SPR) based sensor is proposed and demonstrated for operation in the 1500 nm region. The sensor operates by the immersion of microfibers with a 25-μm diameter waist in chemical solutions that have varying external effective refractive indices. The sensor is fabricated using a homemade single-mode tapered fiber with a uniform waist coated on all sides with a gold layer approximately 45 nm thick by means of a vacuum electron beam evaporator machine. The transmission dips in the sensor’s optical spectrum are highly responsive to any perturbations in the external medium refractive index, thus providing high sensitivity. The proposed sensor system has multiple potential applications in biological and chemical constituent and reaction measurements.

Keywords: Surface Plasmon Resonance (SPR), Microfiber, Gold Coated Tapered Fiber.

1. INTRODUCTION

Optical surface plasmon resonance (SPR)-based sensors systems have attracted considerable attention in recent years⁴⁻⁷ due to the sensitivity of these systems towards perturbations in the refractive index of the surrounding medium’s.⁴⁻⁷ This makes SPR-based sensors systems highly suited towards the applications that involve the monitoring of biological and chemical constituents as well as biomolecular interactions. While SPR-based sensors systems were traditionally bulky in nature, recent advances have seen the development of miniature SPR sensors using metal-coated optical microfibers as a highly potential alternative to the bulk configuration.⁹⁻¹² Fiber-based SPR sensors have multiple advantages over their bulk counterparts, including the capacity for remote deployment, overall lightweight factor and the ability to undergo multiplexing. Optical SPR sensor designs most commonly incorporate D-shaped fibers, uncladded multimode fibers and single-mode tapered fibers.¹³⁻¹⁶ The compact form factor and cost-effectiveness of fiber based SPR optical sensors, combined with recently available compact and more portable optical spectrum analyzers (OSAs) have made this approach the preferred technique for wavelength detection. Of the various fiber based solutions for optical SPR sensors, optical microfibers show the most promise as it is amongst the easiest and cheapest to fabricate. Optical microfibers are fabricated from conventional single mode fibers (SMFs) which are tapered,¹⁷ via heating and stretching,¹⁸ to obtain a waist region that is constant in size and almost similar to the wavelength of the beam that will traverse along its biconical lengthwise shape. Furthermore, by controlling the rate at which the fiber is pulled during its production allows for the tapered fiber’s profile to be fine-tuned towards different applications, thus giving the tapered fiber substantial flexibility.¹⁹ The typical characteristics desired in microfibers themselves are highly desirable for optical sensing applications, including having a large evanescent field,²⁰ strong confinement of the optical signal,²¹ physical flexibility,²²

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easily configurable to multiple applications and system robustness. In addition to this, planar-coated metal layers can also be deposited on the flat side of D-shape SMF, prism, and unclad multimode fibers, thus increasing their potential applications.

SPR-based sensors such as those aforementioned have a metal layer deposited in a planar orientation on the tapered fiber, and will typically exhibit one or more dips in the transmission spectrum. The number of resonance dips depends on the thickness of the metal thin-film; a thinner metal film coating will result in multiple resonance dips being exhibited by a single-mode tapered fiber (SMTF). This is attributed primarily to the thin cylindrical layer of metal supporting hybrid surface plasmon modes. Localized SPR (LSPR) happens when electromagnetic radiation interacts with the electrons in metal-based nanoparticles, such as gold (Au) and silver (Ag), resulting in strong scattering and extinction spectra that can then be exploited for various critical applications.

The LSPR effect can be obtained through the use of various materials, including nanoparticles, nanofibers, nanorods and also nanohole arrays. All these structures show LSPR peaks at different locations, as the LSPR frequency depends primarily on the nanoparticle shape, size and properties of its material as well as the properties of the surrounding medium as explained and discussed in Refs. [31–33].

In this work, the experimental demonstration is given of a uniform-waist SMTF with an even thin gold layer coating that experiences a shift in its transmission spectrum dip when the external medium’s refractive index is altered. The sensitivity of gold-coated microfiber to external medium refractive index variation is analyzed, and a convenient deposition technique (coating) proposed for the fabrication of the SMTF. This effect is demonstrated at the 1.5 μm region, as this region is a common region and can be easily accessed through readily available and low-cost components. The shift is considerably stable and uniform and thus suitable for sensing applications, and the waist diameter of the tapered fiber and its uniformity can be precisely controlled during the tapering process. The high sensitivity of the proposed sensor allows for many practical application, especially those pertaining hazardous environment conditions.

2. THEORETICAL BACKGROUND

The propagation of light within micro- and nano-fiber (MNFs) is made possible due to the differences between the refractive indices of the cladding and core. Similarly, the effective index of the MNF also reduces as the taper waist diameter reduces and as a result of this, the modes propagating in MNFs are not guided by a core-cladding interface, but instead by an air-cladding interface. The propagation of modes within the MNF can be adequately described by Maxwell’s equations, despite a weak propagation approximation in air as follows:

\[
\left[ \begin{array}{c} J'_0(U) + K'_0(U) \\ UJ'_0(U) + WK'_0(U) \end{array} \right] \left[ \begin{array}{c} J'_0(U) + n_{sur}^2 K'_0(U) \\ WUJ'_0(U) + n_{sur}^2 WK'_0(U) \end{array} \right] = \nu^2 \left( \frac{1}{U^2} + \frac{1}{W^2} \right) \left( \frac{1}{U^2} + \frac{1}{W^2} \right) \left( \frac{n_{sur}^2}{U^2} \right) \left( \frac{n_{sur}^2}{W^2} \right) \left( \frac{1}{U^2} + \frac{1}{W^2} \right) (1)
\]

where

\[
U = r \sqrt{k_0^2 n_{MNF}^2 - \beta^2}, \quad W = r \sqrt{\beta^2 - k_0^2 n_{sur}^2} (2)
\]

and \(J_n, K_n\) are the \(n\)th order Bessel functions of the first and second kind respectively. The factors \(n_{MNF}\) and \(n_{sur}\) are the MNF’s and the surrounding medium refractive indices respectively, while \(\beta\) is the optical mode’s effective propagation constant. The core radius, \(r\) matches the radius of the fiber from its center to the cladding-core interface in the case of solid fibers or to the air-cladding interface in the case of MNFs. The hybrid modes’ propagation constant can be obtained through the solution of Eq. (1), and the \(V\) number is given as:

\[
V = \sqrt{U^2 + W^2} = \frac{2\pi}{\lambda} \cdot r \cdot NA (3)
\]

MNFs will operate in the single-mode regime when \(V < 2.405\), and in the case of \(V \ll 1\) a large portion of the power will be propagating as the evanescent field outside the MNF. A significant evanescent field allows for the SPR reflectance, \(R(\theta)\), of a multilayer material to be computed via the materials’ dielectric constants as follows:

\[
R(\theta) = 1 - A(\theta) (4)
\]

where

\[
A(\theta) = \left( \frac{2\pi}{\lambda} \right)^2 \sum_{j=1}^{\infty} \frac{1}{k_p(\theta)} \sum_{j=1}^{N} \int_{r_j}^{r_{j+1}} \text{Im}(\hat{e}_z) \langle E_z^2(\theta) \rangle \, dz (5)
\]

The angle of incidence is denoted by \(\theta\), and \(A(\theta)\) is the absorbance. The complex dielectric constant of the \(j\)th layer is given as \(\hat{\epsilon}\) and the coupled radiation wavelength is given as \(\lambda\). \(\langle E_z^2(\theta) \rangle\) is the mean square evanescent field at a distance of \(z\) from the gold coated microfiber surface. \(N\) is the number gold layers that make the coating of the microfiber, in the sensor setup in which the coated gold over the surface of microfiber, while \(k_p(\theta)\) is the wavevector \(z\)-component of the microfiber and can be described as the wavevector \(x\)-component \(k_p(\theta)\) as follows:

\[
k_p(\theta) = \left( \frac{2\pi}{\lambda} \right)^2 \epsilon_p - k_{p0}^2(\theta) \right)^{1/2} (6)
\]

with

\[
k_{p0}(\theta) = \left( \frac{2\pi}{\lambda} \right)(\epsilon_p \sin^2 \theta)^{1/2} (7)
\]

with the dielectric constant of the microfiber designated as \(\epsilon_p\).
3. EXPERIMENTAL DETAILS

3.1. Fabrication of SMTF

The proposed microfiber is fabricated using a conventional SMF-28 optical fiber with a core diameter of 9 μm and numerical aperture (NA) of 0.14. A custom Mach 3 Computer Numeric Control (CNC) machine was used to stretch the SMF28 to create the taper. This method allows for a tapered fiber with a loss of lower than 0.1 dB and a uniform waist diameter to be fabricated. The structure of the SMTF is given in Figure 1(a). The gradual decrease in the SMTF’s cladding and core diameters will result in the evanescent fields spreading outwards from the core towards the cladding. These evanescent fields can expand beyond the boundary of the SMTF if the waist diameter is narrow enough, whereby the core no longer guides the light propagating in the fiber, and the light is instead now guided by the cladding and external medium. The circular symmetry of the original fiber is still maintained throughout the SMTF despite the tapered diameters. The uniform waist length, \( L \), was 10 mm and the tapered SMTF eventual waist diameter was \( \rho = 25 \) μm in this experiment.

A quasi-circular gold layer with a uniform thickness was coated on the tapered waist area of the microfiber via an Edward Auto 306 Turbo electron beam evaporation system. Figure 1(b) illustrates the process in which a thin layer of gold nanoparticles was evaporated from a solid source and deposited on the sample. For efficient gold coating with a cylindrical thickness of \( t \) on the taper waist, the deposition process needs to be undertaken in three stages wherein the fiber is rotated 120° between each stage. A very small glass substrate was also coated in an identical procedure in order to provide for a measurable indication of the thickness of the layer of gold nanoparticles that now coat the microfiber. Estimating the thickness of the deposited sample requires a scratch to be produced on the sample surface, which was achieved in this work by drawing a straight line using the fine tip of a tweezer. The sample was subsequently placed on the stylus stage of a non-contact profilometer, where thickness measurement allowed for the gold layer deposited onto the microfiber to be measured to be about 45 nm shown in Figure 1(c). The reading of the thickness was plotted as height (Angstrom units) versus distance moved (micrometres), and a huge difference in height was recorded between the non-scratched and scratched regions when the scanner traversed the position of the scratched line. The difference between the resulting height measurements of both regions represented the standard height of the deposited sample. Accurate knowledge relating to the formation of the gold coating is of significant interest due to the crucially related factors such as a suitable refractive index for a particular wavelength range and oxidation resistance as discussed in Ref. [42].

3.2. Measurements

The setup for the SPR measurement system is shown in Figure 2(a). A model AQ2211 tunable laser source (TLS), obtained from Yokogawa was synchronized with a Yokogawa model AQ6370B optical spectrum analyzer (OSA) for optimal simultaneous scanning. This configuration allowed for a laser signal transmission with an accuracy in the order of \( 10^{-3} \) nm and a very sensitive optical detection system for the transmission spectrum scans. Two chemical materials were used in this experiment as external refractive indexes, namely 1-mL diethanolamine (C<sub>4</sub>H<sub>11</sub>NO<sub>2</sub>) and 1-mL glycerol (C<sub>3</sub>H<sub>8</sub>O<sub>3</sub>) with refractive indices of 1.477 and 1.475 respectively.

The thin waist region of the microfiber, approximately 10 mm long was immersed in the index fluid, and the transmission spectrum was recorded while droplets of water, each approximately 0.05 mL were added incrementally to the solution. This gives the transmission spectrum of the proposed system against different external medium refractive indices. Figure 2(b) provides an image of the actual sensor setup, including the uniform-waist SMTF at the center of the image, resting on two fixed stages. A holder with a glass microscope plate lies in the center of the SMTF, upon which the external refractive index solution is added, while at the top of the image the head of

![Fig. 1.](image)

(a) SMTF with uniform waist, in which the final SMTF waist diameter, \( \rho \) is 25 μm an the interaction length, \( L \) is 10 mm. (b) The device cross section, with the shadowed areas representing the coated gold layer with thickness \( t \) of 45 nm. (c) Surface profilometer measurement of gold coated layer thickness.
4. RESULTS AND DISCUSSION

Figure 3(a) shows transmission spectra obtained from the gold nanoparticle-coated SMTF as more water droplet are added to the diethanolamine solution that the SMTF is immersed in, up to a maximum of 15 droplets. The initial dip in the transmission spectrum was observed at 1539 nm, corresponding to pure diethanolamine. The addition of a single water droplet results in the dip shifting slightly to 1538.6 nm as effective refractive index of the solution decreases with the introduction of water, which has a refractive index lower than 1.33. Adding more water droplets to the solution results in the external refractive index lowering further, along with a consequent shift in the transmission dip towards the shorter wavelength. Figure 3(b) shows the shifting of the transmission dips against the changes in the effective refractive index.

It is also important to note that the transmission spectrum in Figure 3(a) exhibits smaller multiple dips, labeled as 2, 3 and 4, which correspond to resonance due to the non-planar and cylindrical geometry of the gold nanoparticle coating on the microfiber. The SPR effect in the planar metal layers are highly TM-polarized, while the cylindrically shaped configuration considered in this work can be viewed as polarization-independent.

Figure 4(a) shows the transmission dips of the gold nanoparticle-coated SMTF when immersed in 1 mL of glycerol (C₃H₈O₃) with up to 10 water droplet increments that changes the external glycerol solution’s effective refractive index. Figure 4(b) displays the wavelength dip values relative to the effective refractive index, where it can be observed that changes are linear against the shifting wavelength steps. Decreasing the refractive index by 0.007 corresponds to a 0.3 nm shift of the dips towards the shorter wavelengths. The sensor was measured to have granularity in the order of 10⁻³ refractive index unit (RIUs) in the range of 1.52 to 1.55 μm.

The trace of Figure 4(a) only shows a single dip mainly due to the limitation of the spectrum scan used. Extending the scan range allows for observation of multiple dips, as can be seen for the case of Diethanolamine.
electron density changes. The sensitivity of the sensor as a function of external effective refractive index is very accurate and commercially. The sensitivity of the sensor as a function of wavelength of the local minima dip.

5. CONCLUSION

An accurate, uniform and linear change in transmission dips in the 1.5 μm optical wavelength region is demonstrated with a gold nanoparticle-coated SMTF based sensor. SPR occurred when light interacts with the gold surface of the SMTF and results in an increase in the effect of evanescent fields. Multiple SPR dips are observed in the transmission spectrum at the 1.5 μm optical wavelength region, which coincides with readily available and cost-effective optical components already available commercially. The sensitivity of the sensor as a function of external effective refractive index is very accurate and monotonic, while the multiple dips provide for extra flexibility to cover more regions in the optical local region. Such as sensor would have high potential to be used in a variety of applications, in particular those involving the measurement of biological and chemical constituents and their reactions.

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References and Notes


5. S. Shukla, N. K. Sharma, and V. Sajal; Sensitivity enhancement of a surface plasmon resonance based fiber optic sensor using ZnO thin film: A theoretical study; Sensors and Actuators B: Chemical 206, 463 (2014).


22. Z. Xu and Z. Zhang; High sensitivity surface plasmon resonance (SPR) refractive index sensor in 1.5 μm; *Ahmad et al.*


24. A. Diez, M. Andrés, and J. Cruz; In-line fiber-optic sensors based on the excitation of surface plasma modes in metal-coated tapered fibers; *Sensors and Actuators B: Chemical* 73, 95 (2001).


32. K.-S. Lee and M. A. El-Sayed; Dependence of the enhanced optical scattering efficiency relative to that of absorption for gold metal nanorods on aspect ratio, size, end-cap shape, and medium refractive index; *The Journal of Physical Chemistry B* 109, 20331 (2005).


