

# A new analysis method towards highly sensitive plasmonic fiber sensors

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*The spectral resonance of surface plasmon (SP) carried by a 50 nm gold layer covering a tilted fiber Bragg grating displays a wide sensitivity to the refractive index (RI) of the surrounding medium. That platform is thus conducive to the development of highly sensitive biosensors. Indeed, the p-polarized electromagnetic field shows a characteristic attenuation of its comb-shaped spectrum. One of the most used demodulation techniques so far used follows a singular mode's evolution at the edge of the attenuation area. The sensitivity of such a technique is linear in a refractive index range limited to  $10^{-3}$  RIU (refractive index unit). This limitation complicates the analysis if the RI spans over two or more domains. In this paper, a new demodulation based on tracking the crossing point between two envelopes of the p-polarized spectrum in the SP attenuation area is presented. This new technique has a double advantage. Firstly, it has an identical sensitivity independent of the refractive index range. Secondly, the sensitivity is more than 20 times higher than the conventional mode method. Furthermore, it is shown that the sensitivity of this method matches the theoretical SP resonance shift prediction.*

## Introduction

Optical fiber is probably one of the most widely used physical platforms for sensor development due to its real-time control, measurement accuracy as well as time-lapse and losses reducing. Besides the telecommunication system, it can be found in various forms as vibration, temperature and pressure sensors. Its resistance to chemical corrosion, flexibility, small size and its reliability have also brought it into the field of biosensors [1]. Within the core of a single-mode optical fiber, a gold-coated tilted fiber Bragg grating (TFBG) provides a promising platform for specific analyte concentration measurement through refractometry. Following a similar principle as in the Kretschmann prism, the TFBG can detect the surrounding refractive index (SRI) change by surface plasmon resonance (SPR) shift. The sensitivity of the widely used demodulation technique is unfortunately not as high as the theoretical prediction for gold-coated TFBGs. In the following, we present a new demodulation method based on tracking a crossing point in the transmission spectrum to confront theoretical predictions with experimental data [2].

## Theoretical concepts

A slightly angled tilted Bragg grating inscribed inside the core of an optical fiber allows coupling between forward propagating core mode to backward propagating cladding mode if the phase-matching condition (1) is fulfilled [3].

$$\lambda_{cl}^i = \left( n_{eff}^{co}(\lambda_{cl}^i) + n_{eff}^{cl}(\lambda_{cl}^i) \right) \Lambda, \quad (1)$$

where  $\Lambda = \Lambda_g / \cos(\theta)$  is the grating period projected along the fiber axis and  $\theta$  the tilt angle.  $n_{eff}^{co} = 1.4447$  and  $n_{eff}^{cl}$  are respectively the effective RIs of the core and cladding at the wavelength of the mode  $i$ . This results in a comb-shaped spectrum that is indicative of the cladding mode resonances. By coating the TFBG with a gold layer of specific thickness, a highly sensitive refractometer for dielectric media can be obtained. This is because a metal-dielectric interface can support non-radiative surface plasmon polariton (most commonly called surface plasmon or SP). Fluctuations in the electron density at the surface of a metal, caused by an incident electromagnetic field, produce an exponentially decreasing electric field in the direction perpendicular to the interface [4]. This type of wave, characteristic of a surface one, is very sensitive to the refractive index of dielectric media. The effective refractive index of such SP is expressed by

$$n_{eff}^{sp} = \sqrt{\frac{\epsilon_m \epsilon_d}{\epsilon_m + \epsilon_d}}, \quad (2)$$

where  $\epsilon_m$  and  $\epsilon_d$  are respectively the permittivity of the metal and the dielectric medium. Thus, if the gold-coated TFBG is immersed in dielectric medium, cladding modes can resonate with SP wavelength and the surface plasmon resonance (SPR) occurs at  $\lambda_{cl}^i$  if  $n_{eff}^{cl}$  is equal to  $n_{eff}^{sp}$ . By inserting (2) into (1), the sensitivity  $d\lambda^{sp}/dn_{SM}$  predicting the evolution of the wavelength of the SPR as a function of the SRI,  $\lambda^{sp}(\epsilon_d)$ , with  $\epsilon_d = n_{SM}^2$ , is

$$\frac{d\lambda^{sp}}{dn_{SM}} = R \left\{ \left. \frac{\partial \lambda^{sp}}{\partial \epsilon_d} \right|_{\epsilon_d = n_{SM}^2} \frac{d\epsilon_d}{dn_{SM}} \right\} = \Lambda R \left\{ \left( \frac{\epsilon_m}{\epsilon_m + n_{SM}^2} \right)^{\frac{3}{2}} \right\}, \quad (3)$$

where  $R\{a\}$  stands for the real part of  $a$ .

## Materials and methods

The optical fiber used in this work is the standard silica telecommunication-grade one (Corning, SMF-28). After a hydrogen-loading process, under  $\sim 200$  bar and  $60^\circ\text{C}$  for 30 hours for photosensitivity enhancements, a 1 cm long  $8^\circ$  tilted Bragg grating is inscribed using the phase mask technique [3] thanks to a 193 nm excimer laser (Noria, from Northlab Photonics). The phase mask has a period of 1100 nm and has been chosen to locate the Bragg wavelength in the spectral domain of interest. After dehydrogenation at  $100^\circ\text{C}$  for 24 hours, a uniform gold layer (characterized by a permittivity  $\epsilon_m = (0.58 - 11i)^2$  [5]) was added by sputtering process (Leica EM SCD 500). The gold-coated TFBG was then immersed in dielectric solutions composed of aqueous LiCl solution (anhydrous, 99%, from Alfa Aesar), a highly soluble salt. Equation (4) in which the LiCl concentration  $C_{LiCl}$  is expressed in g/ml, was used to prepare 35 solutions with

different refractive indices. They were characterized using a portable refractometer (Reichert Analytical instrument, Brix/Ri-check).

$$RI = 0.1963 C_{LiCl} + 1.3332 \quad (4)$$

The refractive index at the wavelength of interest of all solutions was then converted using Sellmeier's dispersion formula [6]. They ranged from 1.3161 (pure water) to 1.3229 with step index of  $2 \cdot 10^{-4}$ . The insertion loss spectra for all the solutions were acquired with an optical vector analyzer (OVA) from Luna and an alignment on the Bragg wavelengths of all the spectra has been performed to get rid of possible parasite fluctuations due to temperature and/or mechanical strain changes.

## Results and discussion

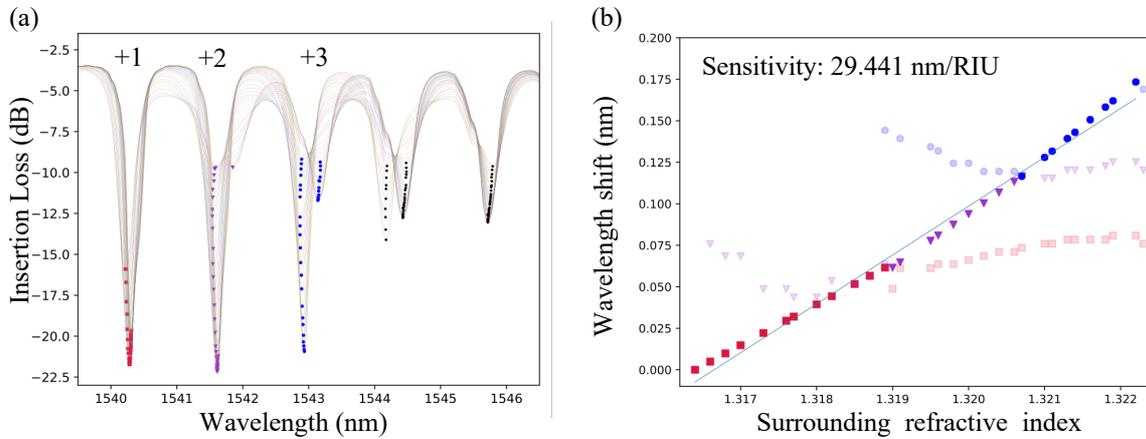


Fig. 1. (a) Insertion loss spectra with a focus around the three modes used in the peaks method, (b) Peaks method evolution showing fiber sensitivity of 29.441 nm/RIU.

The determination of fiber sensitivity to the SRI usually uses the tracking of sensitive modes (namely, +1 to +3, see Fig.1a) between the well-known cut-off mode and the attenuation area. Unfortunately, these modes are sensitive in a limited range of RI as displayed in Fig.1b. The highest sensitivity region of one mode ends where the next one begins. A linear fit on three consecutive modes evolution gives a sensitivity of 29.441 nm/RIU, which is sharply lower than the sensitivity of the theoretical prediction of the plasmonic wavelength shift (567.503 nm/RIU). The new demodulation used hereafter displays a sensitivity twenty times higher than the peak tracking method. It consists in following a specific point in the plasmonic signature. To this aim, the local minimum and local maximum of the upper and lower envelope are first determined [7]. Then, two crossed envelopes around the local extrema are found by cubic spline interpolation and the crossing point is tracked while the SRI changes (see Fig.2a). As shown in Fig. 2b, the wavelength shift of the crossing point is independent of the considered RI range. Moreover, the sensitivity obtained by this method is 569.390 nm/RIU, which almost perfectly corresponds to the theoretical prediction of the SPR shift using (3) with our parameters. Finally, the simplicity of the demodulation method allows fully automated processes. The small difference between both values should stem from the instruments accuracy.

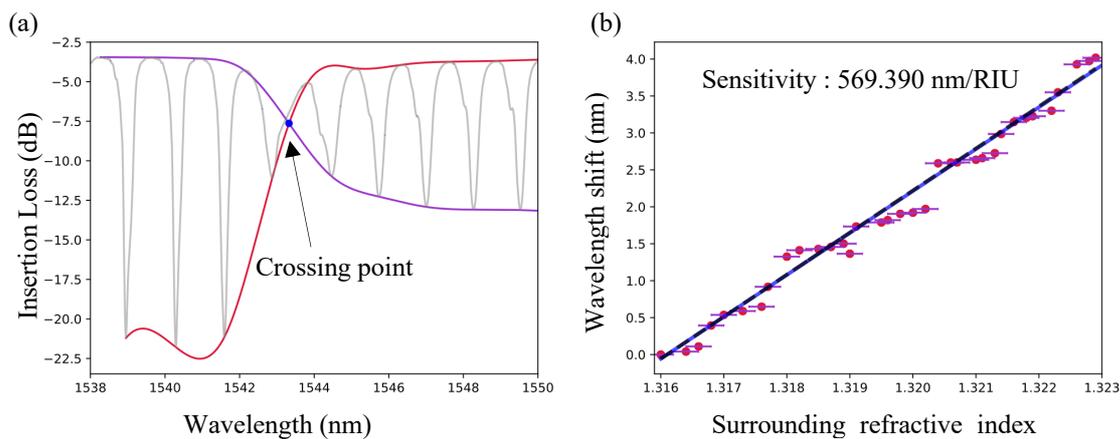


Fig. 2. (a) Crossing point (blue) determination using cross-envelope technic. (b) Wavelength evolution of the crossing points. The measured sensitivity is 569.390 nm/RIU (black dashed line) and theoretical prediction of the SPR sensitivity is 567.503 nm/RIU (blue line).

## Conclusion

Gold-coated TFBGs are promising platforms for the development of highly sensitive biosensors. The demodulation technique based on tracking the crossing point between the two spectral envelopes has shown a sensitivity of 569.390 nm/RIU, which is more than twenty times higher than commonly used one. The simplicity of the method also allows automatization of the demodulation process. Furthermore, similarities between the crossing point sensitivity and the theoretical prediction of the SPR shift were shown. The use of this method on TFBG-based biosensors could lead to the characterization of chemical binding effects in future work.

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## References

- [1] D. J. Monk and D. R. Walt, "Optical fiber-based biosensors," *Anal. Bioanal. Chem.*, vol. 379, no. 7–8, pp. 931–945, 2004, doi: 10.1007/s00216-004-2650-x.
- [2] M. Lobry *et al.*, "Plasmonic fiber grating biosensors demodulated through spectral envelopes intersection," *J. Light. Technol.*, vol. 39, no. 22, pp. 7288–7295, 2021, doi: 10.1109/JLT.2021.3112854.
- [3] J. Albert, L. Shao, and C. Caucheteur, "Tilted fiber Bragg grating sensors," *Laser Photon. Rev.*, vol. 7, no. 4, pp. 83–108, 2012, doi: 10.1002/lpor.201100039.
- [4] S.-V. Berlin, H. New, Y. London, and P. Tokyo, "Heinz Raether Surface Plasmons on Smooth and Rough Surfaces and on Gratings With 113 Figures."
- [5] D. Feng, W. Zhou, X. Qiao, and J. Albert, "High resolution fiber optic surface plasmon resonance sensors with single-sided gold coatings," *Opt. Express*, vol. 24, no. 15, p. 16456, 2016, doi: 10.1364/oe.24.016456.
- [6] M. Daimon and A. Masumura, "Measurement of the refractive index of distilled water from the near-infrared region to the ultraviolet region," *Appl. Opt.*, vol. 46, no. 18, pp. 3811–3820, 2007, doi: 10.1364/AO.46.003811.
- [7] M. Lobry *et al.*, "HER2 biosensing through SPR-envelope tracking in plasmonic optical fiber gratings," *Biomed. Opt. Express*, vol. 11, no. 9, pp. 4862–4871, 2020, doi: 10.1364/boe.401200.