

# Optical fiber immunosensors optimized with cladding etching and ITO nanodeposition

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**Abstract**—Etched optical fiber immunosensors, with and without ITO nanodeposition, have been developed. The performance of these immunosensors has been assessed implementing an immunoassay. The sensitivity of the immunosensor increased by a factor of 4 with the ITO nanocoating, whereas the limit of the detection in both types of devices was 0.2 mg/L of antigens in solution.

**Keywords**—Optical Fiber biosensor; Label-free immunosensors; ITO thin film; E-SMS; IgG/Anti-IgG binding.

## I. INTRODUCTION

The development of real-time detection devices, especially label-free biosensors with high selectivity for the recognition of low concentrations of analytes with rapid response, is an innovative field in applied research and healthcare diagnostics. The single-mode-multimode-single-mode (SMS) optical fiber configuration consists of single-mode fibers (SMFs) spliced to the ends of a section of a multimode coreless fiber (MMF). The operation principle of this refractometer is based on the multimodal interference (MMI). When light propagates along the input SMF and enters the MMF section, several eigenmodes of the MMF are excited and an interference pattern is generated due to the energy exchange among these modes [1].

When the diameter of the MMF section is reduced, the evanescent field of the light in this waveguide penetrates further into the surrounding medium, thus increasing the interaction with this medium and enhancing the sensitivity [2], [3]. In addition, the deposition of a thin film with high refractive index strengthens the interaction with the environment surroundings [3]. The feasibility of Etched SMS (E-SMS) structures as biosensors has been proven recently [4]. The etched structure was able to detect the bound of anti-IgGs to the IgG-based biolayer.

In this work, a thin film of indium tin oxide (ITO) has been deposited in the E-SMS in order to improve sensitivity and study its feasibility as biosensor. Finally, a comparison of both structures has been performed.

## II. METHODS AND MATERIALS

Coreless MMF segments from POFC Inc. and standard SMF pigtails from Telnet Redes Inteligentes Inc. were used. The SMS structure consists of a 14-mm segment of coreless MMF spliced on each end to standard SMF pigtails.

This structure was etched by immersing the fiber in a solution of hydrofluoric acid with a 40% concentration for 50 minutes until the diameter of the fiber was reduced to approximately 25  $\mu\text{m}$ . Then, a thin-film of ITO was deposited by sputtering on the etched region during 60 seconds in a sputtering device (K675XD, Quorum Technologies, Ltd., Sacramento, CA) using a current of 150 mA and a pressure of  $8 \times 10^{-3}$  mbar. The ITO film thickness deposited onto the fiber with these parameters is approximately 40 nm.

Fig. 1 (a) shows the experimental spectra before and after the ITO film deposition. In order to observe the wavelength shift and calculate the sensitivity to refractive index of the surrounding medium, the E-SMS structure before and after the ITO coating was immersed in solutions with various refractive indices [5].

A biolayer was deposited onto the surfaces of the ITO deposited E-SMS to detect the presence of anti-goat IgGs (Sigma Aldrich G4018). The biolayer was composed of a film of IgGs from goat serum (Sigma Aldrich I9140) with a concentration of 11.4 mg/mL. The deposition procedure, solution concentrations, and times involved are detailed in [4].

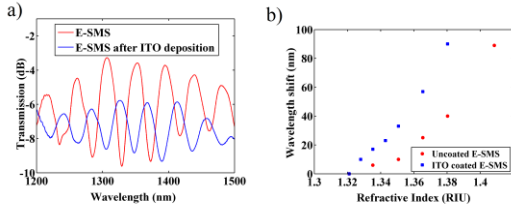


Fig. 1 a) Initial and final transmission spectra due to the ITO thin-film deposition, b) Wavelength shift as a function of refractive for uncoated and ITO coated E-SMS.

### III. RESULTS

Fig 1 (b) illustrates the wavelength shift as a function of the external refractive index, which allows calculating the sensitivity. The wavelength position was taken with the attenuation band closer to 1300 nm. The calculated sensitivity of the ITO coated fiber was 1460 nm/RIU in the 1.333-1.338 RIU range, and 666 nm/RIU in the same range for the uncoated structure.

Following the protocol indicated in [4], the IgG – anti-IgG interaction was measured by increasing the anti-IgG concentration of the testing solution with rinsing stages of the biosensor using PBS solution in between. The PBS rinse stage was used to establish the baseline associated to each increasing anti-goat IgG concentration and to remove the unbound antibodies from the previous solution. Fig. 2 presents the results corresponding to the immunoassay for wavelength tracking of the coated E-SMS when the measurements increased from 1.0 mg/L to 200 mg/L, before the sensor reached saturation.

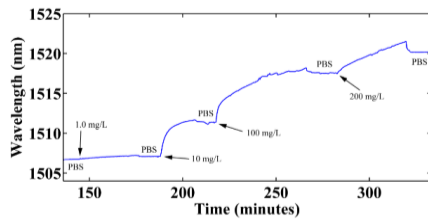


Fig. 2 Sensorgram of the immunoassay with the coated E-SMS.

As aforementioned, the relevant data were collected once the response stabilized in the PBS stages. These values were plotted in Fig. 3 in a logarithmic scale and compared with an immunoassay performed to an uncoated E-SMS. A typical Hill Equation sigmoidal curve has been fitted with a correlation coefficient of 0.9977.

### CONCLUSIONS

Table 1 summarizes the information from the curve fitting with the Hill sigmoidal equation for both bioassays. The ITO coating improves the sensitivity to the Anti-IgG detection. However, the limit of detection (LOD) did not experience any change, because the baseline of the ITO coated device was less stable than the no coated device. This was probably due to the presence of ITO coating. So

further research could be done towards improving this base line and to take advantage of the 4-fold sensitivity improvement obtained with ITO coated E-SMS structure, which indicates this device is an effective and suitable optical platforms for label-free biosensing.

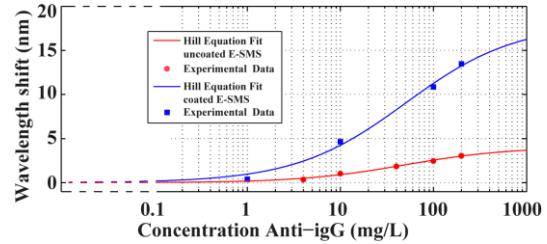


Fig. 3 Calibration curve comparison. The solid lines plot the sigmoidal curve fitting of the experimental data, blue and red for uncoated and coated E-SMS bioprobes respectively.

TABLE 1. PARAMETERS BASED ON HILL FITTING

|                   | <i>Uncoated E-SMS</i> | <i>Coated E-SMS</i> |
|-------------------|-----------------------|---------------------|
| <b>LOD (mg/L)</b> | <b>0.2</b>            | <b>0.2</b>          |
| <b>DSR (nm)</b>   | <b>3.8</b>            | <b>17</b>           |
| <b>WR (mg/L)</b>  | <b>1.5-266</b>        | <b>1.4-516</b>      |

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### REFERENCES

- [1] Q. Wang and G. Farrell, “All-fiber multimode-interference-based refractometer sensor: proposal and design,” *Optics letters*, vol. 31, no. 3, pp. 317–319, 2006.
- [2] J. Lou, L. Tong, and Z. Ye, “Modeling of silica nanowires for optical sensing,” *Optics express*, vol. 13, no. 6, pp. 2135–2140, 2005.
- [3] Y. Cardona-Maya, I. Del Villar, A. B. Socorro, J. M. Corres, I. R. Matias, and J. F. Botero-Cadavid, “Wavelength and Phase Detection Based SMS Fiber Sensors Optimized With Etching and Nanodeposition,” *JOURNAL OF LIGHTWAVE TECHNOLOGY*, vol. 35, no. 17, p. 3743, 2017.
- [4] Y. Cardona-Maya, A. B. Socorro, I. D. Villar, J. L. Cruz, J. M. Corres, and J. F. Botero-Cadavid, “Label-free wavelength and phase detection based {SMS} fiber immunosensors optimized with cladding etching,” *Sensors and Actuators B: Chemical*, vol. 265, pp. 10–19, 2018.
- [5] P. R. Cooper, “Refractive-index measurements of liquids used in conjunction with optical fibers,” *Applied optics*, vol. 22, no. 19, pp. 3070–3072, 1983