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## Sensing Properties of ITO Coated Optical Fibers to Diverse VOCs

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### Abstract

Indium tin oxide ITO-coated optical fibers have been very recently presented as lossy mode resonance (LMR) based refractometers as well as a label-free optical fiber sensing platform. Here, ITO coated optical fiber devices are used for the detection of volatile organic compounds (VOCs). These devices are immune to optical power variations due to their wavelength-based detection technique. More precisely, the sensitivity of these devices to fixed concentrations of ethanol, acetone and methanol has been studied. Furthermore, the cross-sensitivity with temperature and relative humidity (RH) has been addressed.

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*Keywords:* Resonance wavelength, optical fiber, sensors, refractive index, indium tin oxide, volatile organic compounds

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### 1. Introduction

In the last decades transparent conductive oxides (TCOs) have found many applications in different markets from flat-screens or electrochromic windows to solar cells [1]. Among them, the optical and electrical properties of ITO thin-films, have motivated many different works in the last decades [2-3]. Moreover, ITO coated optical fibers have been used in the fabrication of optical fiber sensors [4-5] and, very recently, described as lossy mode resonance (LMR) based refractometers in the infra-red region [6-7]. These devices permit the detection of the refractive index of a sample located onto the ITO film in the same manner as surface plasmon resonance (SPR) based refractometers. LMR-based devices can overcome SPR ones in several aspects, such as the independence with the polarization of light or the presence of multiple resonance peaks by adjusting the LMR supporting coating thickness as it has been discussed in previous works of our group [6-8]. Furthermore, the deposition of additional sensitive coatings onto the ITO layer permit the development of many different sensors [8], in the same way as SPR-based sensors do [9-10]. Additionally, the characteristic conductive properties of ITO thin films permit the detection of the presence of different gases or VOCs [11]. Therefore, this work is aimed to study the spectral response of these LMR-based ITO-coated optical fiber devices to diverse VOCs, such as methanol, ethanol and acetone as well as their cross-sensitivity with temperature or relative humidity (RH) variations.

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## 2. Experimental

### 2.1. Sensor Fabrication

All chemicals were purchased from Aldrich Inc. and used without further purification. A 0.1 M ethanol based solution from indium and tin chloride was prepared. The ratio of In:Sn ions used was 90:10. ITO coatings were fabricated onto the optical fiber core via a dip-coating deposition process described by Ota et al [12]. The fabrication process basically consisted of a prior removal of the cladding from a portion of 4 cm of a 200/215 core/cladding diameter optical fiber (FT200EMT, from Thorlabs Inc). Then, the optical fiber was cleaned under sonication in detergent, ultrapure water and acetone for 10 min. consecutively. After that, the prepared optical fiber substrates were immersed and withdraw from the solution at a constant speed rate (4 cm/s) in order to deposit a single layer. This process was repeated up to the desired thickness or number of layers in order to obtain a 220 nm coating (see Fig. 1). An annealing process was performed at 500 °C during 30 min between each layer deposition and followed by a final annealing step for 4 hours under controlled atmosphere.

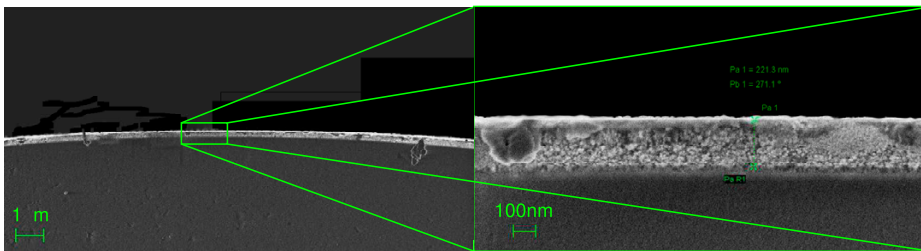


Fig. 1: SEM image and detail of the ITO coated optical fiber

### 2.2. Sensor Characterization

After the fabrication, the ITO coated optical fibers were cleaved and spliced to optical fiber patch cords at both ends. One end was connected to a broad spectrum halogen white light source (Ando AQ4303B) and the other to a HR4000 spectrometer (Oceanoptics Inc.) as it is represented in Fig. 2. The sensing setup basically consisted in an optical transmission configuration where the light enters into the optical fiber, travels through the sensitive region and is collected at the end of the optical fiber. Additionally, it was used an environmental chamber and a sealed box in order to measure the sensitivity of the device to temperature, relative humidity and volatile organic compounds (VOCs). All the experiments were performed at room conditions (20 °C and 30% RH) unless otherwise stated.

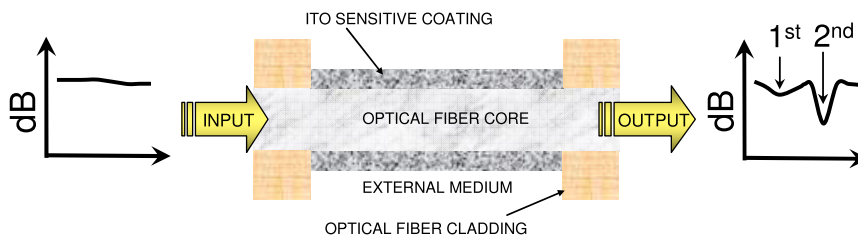


Fig. 2: Schematic representation of the optical fiber sensitive region and sensing mechanism.

### 3. Results and Discussion

As it has been shown in previous works of our group, ITO coated optical fibers act as resonance-based refractometers in the infra-red region [7] showing one or more resonances as a function of the ITO coating thickness [6]. Thus, part of the light passing through the sensitive region (the region with the ITO coating) is absorbed originating one or more resonances, which are sensitive to variations of the refractive index of the external medium in contact with the ITO layer.

The spectral response of the fabricated device showed two differentiated resonance peaks located at 618 nm (first peak) and 874 nm (second peak) respectively when it was excited by a halogen white light source as it is schematically represented in Fig. 2. Then, in order to test the sensitivity of the device to different VOCs, the sensitive region of the device was placed into a sealed box and exposed to a concentration of 500 ppm of ethanol, methanol and acetone. The wavelength shift of each resonance peak is shown in Table 1 for each of the VOCs separately. The device shows higher sensitivity to ethanol with an average shift of 26 nm, which agrees with previous studies performed with ITO [11].

Table 1: Resonance wavelength shift of both resonance peaks when the sensitive region is exposed to different VOCs (500 ppm) at room conditions (20 °C and 30% RH).

	C <sub>2</sub> H <sub>5</sub> OH (ethanol)	CH <sub>3</sub> OH (methanol)	C <sub>3</sub> H <sub>8</sub> O (acetone)
Shift of 1st Resonance Peak (nm)	+25.7	+11.2	+5.6
Shift of 2nd Resonance Peak (nm)	+26.5	+12.8	+8.4

Additionally, the sensitivity of the device was tested for variations in the RH from 20% to 80% (see Fig. 3) and temperature from 20 °C to 80 °C (see Fig. 4). In Fig. 3a it is represented the spectral response of the device when the sensitive region is subjected to a variation from 20% to 80% of RH conditions. The shift of the resonance wavelength as a function of the RH is represented in Fig. 3b, showing a considerable drift ( 17nm) of both resonance peaks which suggests the necessity of a RH control in order to perform accurate VOC concentration measurements. On the other hand, temperature variations showed a very small ( 2nm) shift of both resonance wavelengths as it is represented in Fig. 4b, which permit the utilization of the device in a wide range of temperatures without additional requirements.

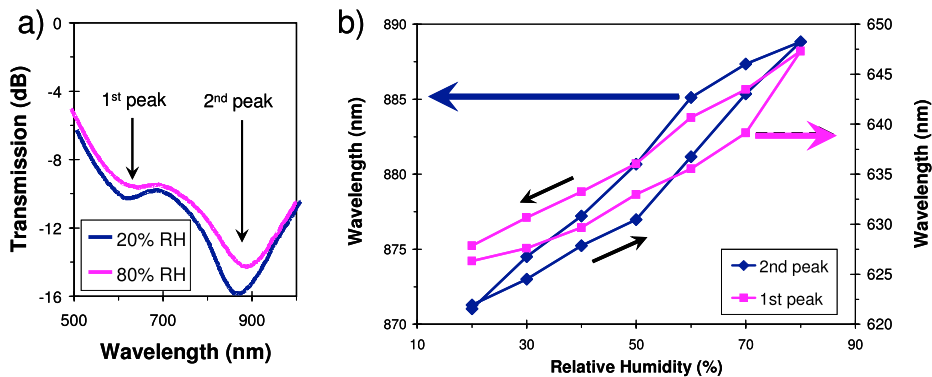


Fig. 3: (a) Spectral response at 20% and 80% RH. (b) Wavelength shift of both resonance peaks from 20% to 80% RH. Measurements performed at constant temperature (20°C).

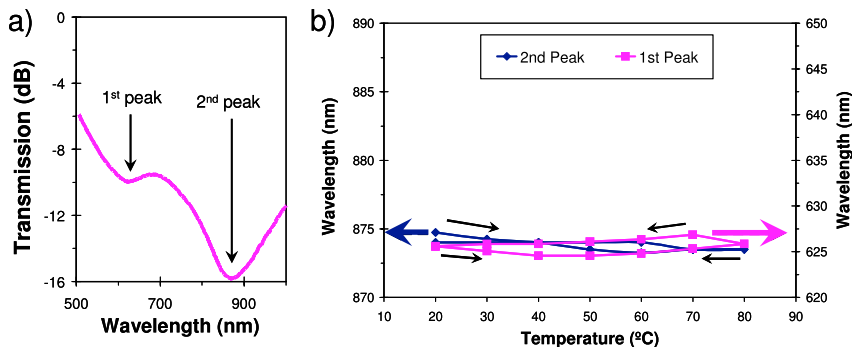


Fig. 4: (a) Spectral response of the sensor at 30 °C. (b) Wavelength shift of both resonance peaks from 20 °C to 80 °C. Measurements performed at constant RH (30%).

#### 4. Conclusions

ITO coated optical fiber devices based on LMRs have been proven as suitable candidates for VOCs concentration measurement. However, a more profound study is required in order to determine the optimum working point of the device and the sensitivity of the device to different VOCs.

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#### References

- [1] Ginley DS, Bright C. Transparent Conducting Oxides. *MRS BULLETIN* 2000;15-18.
- [2] Alam MJ, Cameron DC. Optical and electrical properties of transparent conductive ITO thin films deposited by sol-gel process. *Thin Solid Films* 2000;00:455-459.
- [3] Zamarreño CR, Goicoechea J, Matias IR, Arregui FJ. Laterally selective adsorption of pH sensing coatings based on neutral red by means of the electric field directed layer-by-layer self assembly method. *Thin Solid Films* 2009;517:3776-3780.
- [4] Konry T, Marks RS. Physico-chemical studies of indium tin oxide-coated fiber optic biosensors. *Thin Solid Films* 2005;492:312-321.
- [5] Larrion B, Hernaez M, Arregui FJ, Goicoechea J, Bravo J, Matias IR. Photonic Crystal Fiber Temperature Sensor Based on Quantum Dot Nanocoatings. *Journal of Sensors* 2009;2009:932471
- [6] Del Villar I, Zamarreño CR, Hernaez M, Matias IR, Arregui FJ. Lossy mode resonance generation with indium tin oxide coated optical fibers for sensing applications. *J Lightwave Tech* 2010;28:111-117.
- [7] Zamarreño CR, Hernaez M, Del Villar I, Matias IR, Arregui FJ. ITO coated optical fiber refractometers based on resonances. *IEEE Sensors Journal* 2010;10:365-366.
- [8] Zamarreño CR, Hernaez M, Del Villar I, Matias IR, Arregui FJ. Tunable humidity sensor based on ITO-coated optical fiber. *Sensors and Actuators B* 2010;146:414-417.
- [9] Homola J, Yee SS, Gaughlitz G. Surface plasmon resonance sensors: review. *Sensors and Actuators B* 1999;54: 3-15
- [10] Gupta BD, Verma RK. Surface Plasmon Resonance-Based Fiber Optic Sensors: Principle, Probe Designs, and Some Applications. *Journal of Sensors* 2009;2009: 979761
- [11] Jiao Z, Wu M, Gu J, Sun X. The gas sensing characteristics of ITO thin film prepared by sol-gel method. *Sensors and Actuators B* 2003;94:216-221.
- [12] Ota R, Seki S, Ogawa M, Nishide T, Shida A, Ide M, Sawada Y. Fabrication of Indium-tin-oxide films by dip coating process using ethanol solution of chlorides and surfactants. *Thin Solid Films* 2002;411:42-45.