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# Wavelength and Phase Detection Based SMS Fiber Sensors Optimized With Etching and Nanodeposition

Yamile Cardona-Maya, Ignacio Del Villar, Abian B. Socorro, Jesus M. Corres, *Member, IEEE*, Ignacio R. Matias, *Senior Member, IEEE*, and Juan F. Botero-Cadavid

Abstract—The development of an optical fiber refractometer by 5 hydrogen fluoride etching and sputtering deposition of a thin-film 6 of indium tin oxide on a single-mode-multimode-single-mode fiber 7 structure has been analyzed with the aim of improving the sen-8 9 sitivity to the changes of the refractive index (RI) of the external medium. The device is sensitive to the RI changes of the surround-10 ing medium, which can be monitored by tracking the spectral 11 changes of an attenuation band or with a fast Fourier transform 12 (FFT) analysis. By using an optical spectrum analyzer combined 13 with a simple FFT measurement technique, the simultaneous real-14 time monitoring is achieved. The results show that the sensitivity 15 depends on the thin-film thickness. A maximum of 1442 nm/RIU 16 (refractive index unit) in the 1.32–1.35 RIU range has been attained. 17 In addition, a theoretical analysis has been performed, where simu-18 lations matched with the experimental results. As a practical appli-19 cation of the developed optical fiber structure, a °Brix (°Bx) sensor 20 has been implemented with a sensitivity of 2.13 nm/°Bx and 0.25 21 rad/°Bx respectively for wavelength and phase shift detection. 22

*Index Terms*—Etching, optical fiber sensor, refractive index,
 single-mode–multimode–single-mode (SMS), thin-films.

### I. INTRODUCTION

PTICAL fiber refractometers have been extensively studied for chemical, medicine, and biological applications due their multiple advantages such as compact size and high resolution. They can be used in harsh environments and allow for minimally invasive procedures to be performed [1].

Up until now, several technologies have been used to develop optical fiber refractometers. Some of these technologies include: fiber gratings [2], [3], long period fiber gratings [4], resonances [5], [6], evanescent field [7] and interferometers [8].

Manuscript received April 21, 2017; revised June 5, 2017; accepted June 16, 2017. This work was supported in part by the Agencia Estatal de Investigación, in part by Fondo Europeo de Desarrollo Regional (TEC2016-78047-R), in part by the Government of Navarre through its projects with references: 2016/PI008, 2016/PC025, and 2016/PC026, and in part by the Colombian Administrative Department of Science, Technology and Innovation—Colciencias, through the Program for national doctorates, calling 617 of 2013. (*Corresponding author: Yamile Cardona-Maya.*)

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Digital Object Identifier 10.1109/JLT.2017.2719923

Within the last group, a single-mode-multimode-single-mode 35 SMS fiber refractometer has been studied and a sensitivity of 36 1199.8 nm / RIU for the range of refractive indices from 1.321 37 to 1.382 has been reported [9]. In this work, the experimental 38 and theoretical demonstration of a novel and high-sensitivity re-39 fractometric sensor is reported. The developed sensor relies on 40 multimodal interference in etched SMS fiber structures coated 41 with a thin-film that improves the previously sensitivity reported 42 with this structure [9]. Moreover, by application of fast Fourier 43 transform analysis (FFT), it is possible to use both wavelength 44 and phase monitoring of the parameter to detect. 45

The proposed SMS configuration of this work consists of 46 input and output single-mode fibers (SMFs) that are spliced 47 to a section of a multimode coreless fiber (MMF) of a certain 48 length. The operation mechanism of this refractometer is based 49 on the multimode interference (MMI). When the light propagat-50 ing along the input SMF enters the MMF section, several eigen-51 modes of the MMF are excited and interference among different 52 modes occurs during the propagation along the MMF section. 53 Finally, the light is coupled into the output SMF at the end of 54 the MMF section [10]. Through the reduction of the diameter 55 in the MMF section, the evanescent field penetrates further into 56 the surrounding medium, thus increasing the sensitivity [11], 57 [12]. Furthermore, a deposited high refractive index thin-film 58 enhances the interaction with the environment surrounding the 59 fiber and also permits to increase the sensitivity. 60

To sum up, the enhancement of the sensitivity in the SMS configuration in this work is achieved by gathering two different phenomena, diameter reduction and thin-film deposition. 63

#### II. METHODS AND MATERIALS

Coreless MMF segments from POFC Inc. (Taiwan) and 65 standard SMF pigtails from Telnet Redes Inteligentes Inc. 66 (Zaragoza, Spain) were used in this study. The SMS structure 67 consists on a 14-mm segment of coreless MMF spliced on each 68 end to standard SMF pigtails, as shown in Fig. 1(a). This struc-69 ture was etched using hydrofluoric acid 40% (HF) until the 70 diameter of the fiber was reduced to approximately 25  $\mu$ m [see 71 Fig. 1(b)]. This process took 70 minutes. Finally, a thin-film 72 of Indium Tin Oxide (ITO) was deposited by sputtering on the 73 etched region [see Fig. 1(c)]. The effect of the etching and the 74 deposition was studied theoretically and experimentally. 75

The theoretical analysis was performed with FIMMWAVE. 76 The propagation was obtained with FIMMPROP, a module in- 77

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Fig. 1. (a) 14-mm segment of coreless MMF spliced on each end to standard SMF forming an SMS structure, (b) SMS structure after the HF etching, (c) etched SMS structure with an ITO thin-film deposition.

tegrated with FIMMWAVE. Finite difference method FDM was
used for the SMF and MMF sections, since it is the most accurate method available for cylindrical waveguides. In the SMF
sections only the fundamental mode was analyzed, whereas for
the MMF section 30 modes were analyzed, thus allowing to
achieve convergence in the results.

### 84 A. Diameter Reduction

Light from a white SLED source was launched through the SMS structure during the etching stage in a 40% HF solution until the diameter of the fiber was reduced to approximately 25  $\mu$ m. The transmission spectrum was recorded by an Optical Spectrum Analyzer (OSA). Fig. 2 depicts the experimental setup.

#### 90 B. Film Deposition

Three SMS structures were etched with the aforementioned procedure, hereafter called Sensor 1, Sensor 2, and Sensor 3,



Fig. 2. Experimental setup for etching the SMS structure.



Fig. 3. Ellipsometry analysis of the ITO film used in this work.

respectively. Light from a white SLED source was launched into the structures while an ITO thin-film was deposited by sputtering (first on Sensor 2, and then on Sensor 3). An Optical Spectrum Analyzer (OSA) recorded the transmission spectra during the deposition. 97

Sensor 1 had no thin-film, Sensor 2 and Sensor 3 were deposited with ITO during 45 and 75 seconds respectively in a sputtering device (K675XD from Quorum Technologies, Ltd.) 100 using 150 mA current and  $8 \times 10^{-3}$  mbar pressure. 101

To study the effect of the ITO thin-film deposition theoretically, it was necessary to obtain its dispersion curves. Fig. 3 depicts the ellipsometry analysis performed. This ellipsometric information allowed to compare the theoretical wavelength spectra before and after the deposition.

### C. Device Characterization by Wavelength Shift

After the fabrication of the sensors, the same setup shown in 108 Fig. 2 was used to characterize them when subjected to changes 109 in the external RI. In order to observe the wavelength shift, the 110 sensitive structure was immersed in various solutions of glycerol 111 in water at different concentrations [13], [14]. The sensitivity 112 curves were studied by tracking the spectral changes of the 113 nearest attenuation band to a wavelength of 1550 nm. 114

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This characterization was performed to all three sensors fabricated: Sensor 1, Sensor 2, and Sensor 3.

Theoretically, the sensor characterization was performed by 117 simulating with FIMMWAVE the SMS structure after the etching procedure for the three film conditions proposed, for different refractive indices of the surrounding medium. The refractive 120



Fig. 4. Amplitude of the fast Fourier transform before (a) and after (b) of the theoretical spectrum obtained by etching.

121 index of the optical fiber cladding, made of fused silica, was es-122 timated by using the Sellmeier equation:

$$n^{2}(\omega) = 1 + \sum_{j=1}^{m} \frac{B_{j}\omega_{j}^{2}}{\omega_{j}^{2} - \omega^{2}}$$
(1)

with parameters: B1 = 0.691663, B2 = 0.4079426, B3 = 0.8974794,  $\lambda 1 = 0.0684043 \,\mu m$ ,  $\lambda 2 = 0.1162414$ , and  $\lambda 3 =$ 9.896161, where  $\lambda j = 2\pi c/\omega j$  and c is the speed of light in vacuum [15]. The optical fiber core refractive index for the simulations was obtained, according to the specifications from the fiber manufacturer, by increasing the refractive index of the cladding 0.36%.

# D. Degrees Brix (°Bx) Sensor Monitored by Both Wavelength and Phase Shift Detection

Typically, sensors are characterized by tracking the wave-132 length of an attenuation band using an optical spectrum an-133 alyzer, or alternatively by measuring the intensity variations 134 at a fixed wavelength using a power meter. The fast Fourier 135 transform (FFT) analysis, which permits to extract the phase of 136 the optical spectrum, is not a broadly used technique despite 137 it provides useful and clear information to be used in sensing 138 applications and permits to use interrogators instead of optical 139 spectrum analyzers [16], [17]. 140

The sinusoidal spectrum of the SMS sensors after etching permits to see a sharp peak corresponding to the fundamental frequency (see in Fig. 4 the comparison between the magnitude of the fast Fourier transform before and after etching). Consequently, it is possible to obtain a phase sensitive device by tracking the phase of this fundamental frequency as a function of the parameter to detect.

In order to probe the feasibility of this method for the developed sensor shown here, a degrees Brix sensor by phase shift detection is presented. The ITO thickness film to the sensor used in this application is approximately 60 nm. A MATLAB



Fig. 5. Evolution of the wavelength spectrum due to reduction of the diameter by HF etching: (a) Experimental; (b) theoretical.

script was implemented to obtain the phase of the fundamental 152 frequency in the optical spectrum response of the sensor.

Degrees Brix is a scale of relative density used in the sugar and winemaking industry. It indicates the percentage of cane sugar by weight in a solution or juice of unfermented grapes. Its measurement is crucial in many applications, such as fruit juice, carbonated beverage industry, and wine making.

The solutions used here were prepared by dissolving sucrose 159 in distilled water. One (1) °Bx equals one (1) gram of sucrose 160 dissolved in 100 grams of solution. 161

## E. Phase Shift and Temperature Cross Sensitivity

A 40-nm thickness ITO thin-film deposited sensor fabricated 163 following the same procedure described for Sensor 2 was placed 164 in a water cell with temperature control. The temperature was 165 set to 40 °C and the spectra started being recorded after reaching 166 this set point, for 10 minutes. After this time, the control was set 167 to 30 °C and kept at this constant temperature for 20 minutes. 168 Finally, the temperature control was set back at 40 °C for 10 169 minutes once this temperature was reached. The real time phase 170 shift was recorded and processed along with the entire procedure 171 of temperature variation. 172

## A. Diameter Reduction

The experimental and theoretical evolution of the wavelength 175 spectrum as a function of the fiber diameter are depicted in 176 Fig. 5(a) and (b) respectively. 177

Fig. 6(a) and (b) show the theoretical and experimental transmission spectra for both, etched and unetched fibers. Video files of this experimental and theoretical evolution can be found in the supplementary material of this manuscript. According to [9], [18], the diameter reduction is proportional to the sensitivity increase to refractive index. Consequently, a reduction from 125 to 25  $\mu$ m should lead to a fivefold increase. 184

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Fig. 6. Comparison between the spectra of unetched and etched fibers: (a) Experimental; (b) theoretical.



Fig. 7. Experimental evolution of the spectra during ITO deposition from: (a) Sensor 2; (b) Sensor 3.

In addition to the structural integrity of the fiber, it was observed that at this diameter the transmission evolved into a quasisinusoidal spectrum for a length of the coreless MMF of 14 mm.
This behavior facilitates both the presence of multiple attenuation bands and the shift phase study.

### 190 B. Film Deposition

Fig. 7(a) and (b) present the spectral response obtained fromSensors 2 and 3 during the ITO films deposition.

For both cases, the transmission spectra underwent redshifts during the deposition, proving the existence of a relationship between the spectral position and the ITO film thickness. Sensor 2 and Sensor 3 exhibited 23 nm and 40 nm redshifts, respectively. Fig. 8(a) and (b), and Fig. 9(a) and (b) show the experimental and theoretical spectra before and after the ITO film deposition for Sensors 2 and 3, respectively.

It can be observed that the experimental redshifts due to the ITO deposition were in essence the same as those obtained theo-



Fig. 8. Initial and final transmission spectra due to the ITO thin-film deposition on Sensor 2: (a) Experimental; (b) theoretical.



Fig. 9. Initial and final transmission spectra due to the ITO thin film deposition on Sensor 3: (a) Experimental; (b) theoretical.

retically. This probes that the transmission spectrum experiences 202 a redshift as thin-film thickness grows. 203

Based on theoretical analysis, it can be concluded that the 204 thicknesses of the deposited thin-film were of 40 nm and 60 205 nm to Sensor 2 and Sensor 3, respectively. To support the good 206 match between the experimental and theoretical results, Sensor 2 207 was cleaved and observed using a scanning electron microscope 208 (SEM). Fig. 10 shows the cross section, where the measure-209 ment of the film thickness was 44.18 nm. This supposes a 10% 210 deviation with respect to the theoretical value for this sensor. 211

#### C. Wavelength Shift Characterization of the Device 212

Figs. 11, 12, and 13 show the theoretical and experimental 213 transmission spectra as a function of wavelength for various 214 refractive indices. A redshift can be observed in all cases when 215 the RI increases, and there is a good agreement between the 216 theoretical and experimental results. 217

Fig. 14 illustrates the wavelength shift as a function of the 218 external refractive index, which allows the sensitivity to be calculated both experimentally [see Fig. 14(a)] and theoretically 220 [see Fig. 14(b)]. In both cases the wavelength position was 221 taken with the attenuation band closer to 1550 nm. 222



Fig. 10. SEM images of Sensor 2: Diameter 25  $\mu$ m and ITO thin film 44 nm.



Fig. 11. Transmission spectrum for Sensor 1 as a function of the wavelength for different surrounding media refractive indices: (a) Experimental; (b) theoretical.

It can be noticed that an increase in the thin-film thickness leads to a higher sensitivity. Table I shows a summary of the sensitivities in the 1.32–1.35 refractive index range. The experimental sensitivity improves that obtained in [9]. Indeed, even though a sensitivity of 1200 nm/RIU was attained in that work, the SRI range was 1.32–1.38, with a higher sensitivity. More-



Fig. 12. Transmission spectrum for Sensor 2 as a function of the wavelength for different surrounding media refractive indices: a) Experimental; b) theoretical.



Fig. 13. Transmission a) experimental and b) theoretical for Sensor 3 as a function of the wavelength for different surrounding media refractive indices.



Fig. 14. Wavelength shift with refractive index to Sensor 1, 2 and 3, for both, a) experimental and b) theoretical cases.

TABLE I Comparison Sensitivities Obtain Experimentally and Theoretically to A 1.32–1.35 RIU Range

Thin-film Thickness (nm)	Experimental Sensitivity	Theoretical Sensitivity
Uncoated	335 nm/RIU	454 nm/RIU
	eee militee	ie i mili ide
	$R^2=0.9812$	$R^2=0.9669$
~ 40	1062 nm/RIU R <sup>2</sup> =0.9906	1131 nm/RIU R <sup>2</sup> =0.999
$\sim 60$	1442 nm/RIU R <sup>2</sup> =0.953	1536 nm/RIU R <sup>2</sup> =0.9951



Fig. 15. Transmission as a function of the wavelength to different ° Bx of an (a) etched SMS configuration without thin-film and (b) a 60 nm thin-film Thickness ITO etched SMS configuration.

over, though overcome by LPFGs optimized with a hard etching
[19], the device presented here is comparable with the sensitivity
obtained with LPFGs optimized with a soft etching [20], which
along with the possibility to monitor the phase shift indicates
that it is an interesting device for biosensing applications, where
a high degree of accuracy.

# D. Degrees Brix (°Bx) Sensor Monitored by Both Wavelength and Phase Shift Detection

Fig. 15 shows transmission as a function of the wavelength for different solutions of sucrose in water addressing two cases: an etched SMS configuration without thin-film and a 60 nm thickness ITO thin-film deposited on it. Both cases show a redshift when the probe was immersed in.

The magnitude of the wavelength shift is more notorious in the coated fiber, regardless of the attenuation observed, according to what has been observed in the previous section.

Fig. 16(a) and (b) present the wavelength shift of an attenuation band and the phase shift for both the etched SMS configuration without ITO coating and the same configuration type with a 60 nm thickness ITO thin-film. A simple linear fit of the results evidences that the ITO film permitted to obtain a three-fold sensitivity increase in both cases.



Fig. 16. Phase shift of a brix grades sensor: (a) Without ITO thin-film; (b) with 60 nm thickness ITO thin-film.



Fig. 17. (a) Wavelength shift and (b) phase shift temperature cross sensitivity.

#### E. Temperature Cross Sensitivity

Fig. 17(a) and (b) show the behavior of the wavelength shift 252 of an attenuation band and the phase shift with the temperature 253 setting as described in Section II-E. This probe was realized 254 with the 60 nm thin-film thickness sensor. The phase behavior 255 observed followed a trend imposed by the temperature's profile 256 generated as a consequence of the set points established. The 257 sensitivity was 0.3 nm/°C and 0.034 rad/°C. 258

A 0.14 °Bx/°C of sensitivity was calculated for the ITO thinfilm coated sensors analyzed in the previous section. 260

This manuscript presented the optimization of SMS fiber 262 structures with a combined application of two techniques: etching and deposition of a thin-film. By an adequate design it 264 was possible to track both wavelength shifts of the optical spectrum and, by applying a simple FFT measurement technique, the 266 phase shift of the fundamental frequency. The FFT measurement

used in the analysis is an easy method that can be most applicable 268 to networks that require narrow band, multiplexing capability 269 270 and that have some problems related with high losses and noise.

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The results also showed that the sensitivity obtained for this 271 configuration of SMS was enhanced by reduction of the fiber 272 diameter and by increasing the ITO film thickness. A good 273 agreement was achieved between the experimental and the sim-274 ulated approaches for this sensing device. A sensitivity of 1442 275 276 nm/RIU was obtained by tracking the wavelength shift in a SMS with 25  $\mu$ m diameter and a 60 nm ITO thickness film, whereas 277 for the same device, the FFT phase shift analysis showed a 278 0.24 rad/RIU sensitivity. 279

These sensitivities, which are in the order of magnitude of 280 other structures such as long period fiber gratings (LPFGs) (but 281 with an inherently simpler manufacture process), place the de-282 veloped sensing device as a good option for applications where 283 high sensitivities and compact structures are required. As an 284 example, a degrees Brix sensor has been presented, where the 285 deposition of an ITO thin-film enhances the sensitivity of the 286 287 device by a factor of 3.

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Manuscript received April 21, 2017; revised June 5, 2017; accepted June 16, 2017. This work was supported in part by the Agencia Estatal de Investigación, in part by Fondo Europeo de Desarrollo Regional (TEC2016-78047-R), in part by the Government of Navarre through its projects with references: 2016/PI008, 2016/PC025, and 2016/PC026, and in part by the Colombian Administrative Department of Science, Technology and Innovation—Colciencias, through the Program for national doctorates, calling 617 of 2013. (*Corresponding author: Yamile Cardona-Maya.*)

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Digital Object Identifier 10.1109/JLT.2017.2719923

Within the last group, a single-mode-multimode-single-mode 35 SMS fiber refractometer has been studied and a sensitivity of 36 1199.8 nm / RIU for the range of refractive indices from 1.321 37 to 1.382 has been reported [9]. In this work, the experimental 38 and theoretical demonstration of a novel and high-sensitivity re-39 fractometric sensor is reported. The developed sensor relies on 40 multimodal interference in etched SMS fiber structures coated 41 with a thin-film that improves the previously sensitivity reported 42 with this structure [9]. Moreover, by application of fast Fourier 43 transform analysis (FFT), it is possible to use both wavelength 44 and phase monitoring of the parameter to detect. 45

The proposed SMS configuration of this work consists of 46 input and output single-mode fibers (SMFs) that are spliced 47 to a section of a multimode coreless fiber (MMF) of a certain 48 length. The operation mechanism of this refractometer is based 49 on the multimode interference (MMI). When the light propagat-50 ing along the input SMF enters the MMF section, several eigen-51 modes of the MMF are excited and interference among different 52 modes occurs during the propagation along the MMF section. 53 Finally, the light is coupled into the output SMF at the end of 54 the MMF section [10]. Through the reduction of the diameter 55 in the MMF section, the evanescent field penetrates further into 56 the surrounding medium, thus increasing the sensitivity [11], 57 [12]. Furthermore, a deposited high refractive index thin-film 58 enhances the interaction with the environment surrounding the 59 fiber and also permits to increase the sensitivity. 60

To sum up, the enhancement of the sensitivity in the SMS 61 configuration in this work is achieved by gathering two different 62 phenomena, diameter reduction and thin-film deposition. 63

#### II. METHODS AND MATERIALS

Coreless MMF segments from POFC Inc. (Taiwan) and 65 standard SMF pigtails from Telnet Redes Inteligentes Inc. 66 (Zaragoza, Spain) were used in this study. The SMS structure 67 consists on a 14-mm segment of coreless MMF spliced on each 68 end to standard SMF pigtails, as shown in Fig. 1(a). This struc-69 ture was etched using hydrofluoric acid 40% (HF) until the 70 diameter of the fiber was reduced to approximately 25  $\mu$ m [see 71 Fig. 1(b)]. This process took 70 minutes. Finally, a thin-film 72 of Indium Tin Oxide (ITO) was deposited by sputtering on the 73 etched region [see Fig. 1(c)]. The effect of the etching and the 74 deposition was studied theoretically and experimentally. 75

The theoretical analysis was performed with FIMMWAVE. 76 The propagation was obtained with FIMMPROP, a module in- 77

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(c)

Fig. 1. (a) 14-mm segment of coreless MMF spliced on each end to standard SMF forming an SMS structure, (b) SMS structure after the HF etching, (c) etched SMS structure with an ITO thin-film deposition.

tegrated with FIMMWAVE. Finite difference method FDM was
used for the SMF and MMF sections, since it is the most accurate method available for cylindrical waveguides. In the SMF
sections only the fundamental mode was analyzed, whereas for
the MMF section 30 modes were analyzed, thus allowing to
achieve convergence in the results.

### 84 A. Diameter Reduction

Light from a white SLED source was launched through the SMS structure during the etching stage in a 40% HF solution until the diameter of the fiber was reduced to approximately 25  $\mu$ m. The transmission spectrum was recorded by an Optical Spectrum Analyzer (OSA). Fig. 2 depicts the experimental setup.

#### 90 B. Film Deposition

Three SMS structures were etched with the aforementioned procedure, hereafter called Sensor 1, Sensor 2, and Sensor 3,



Fig. 2. Experimental setup for etching the SMS structure.



Fig. 3. Ellipsometry analysis of the ITO film used in this work.

respectively. Light from a white SLED source was launched into the structures while an ITO thin-film was deposited by sputtering (first on Sensor 2, and then on Sensor 3). An Optical Spectrum Analyzer (OSA) recorded the transmission spectra during the deposition. 97

Sensor 1 had no thin-film, Sensor 2 and Sensor 3 were deposited with ITO during 45 and 75 seconds respectively in a sputtering device (K675XD from Quorum Technologies, Ltd.) 100 using 150 mA current and  $8 \times 10^{-3}$  mbar pressure. 101

To study the effect of the ITO thin-film deposition theoretically, it was necessary to obtain its dispersion curves. Fig. 3 depicts the ellipsometry analysis performed. This ellipsometric information allowed to compare the theoretical wavelength spectra before and after the deposition.

#### C. Device Characterization by Wavelength Shift

After the fabrication of the sensors, the same setup shown in 108 Fig. 2 was used to characterize them when subjected to changes 109 in the external RI. In order to observe the wavelength shift, the 110 sensitive structure was immersed in various solutions of glycerol 111 in water at different concentrations [13], [14]. The sensitivity 112 curves were studied by tracking the spectral changes of the 113 nearest attenuation band to a wavelength of 1550 nm. 114

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This characterization was performed to all three sensors fabricated: Sensor 1, Sensor 2, and Sensor 3.

Theoretically, the sensor characterization was performed by 117 simulating with FIMMWAVE the SMS structure after the etching procedure for the three film conditions proposed, for different refractive indices of the surrounding medium. The refractive 120

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Fig. 4. Amplitude of the fast Fourier transform before (a) and after (b) of the theoretical spectrum obtained by etching.

121 index of the optical fiber cladding, made of fused silica, was estimated by using the Sellmeier equation:

$$n^{2}(\omega) = 1 + \sum_{j=1}^{m} \frac{B_{j}\omega_{j}^{2}}{\omega_{j}^{2} - \omega^{2}}$$
(1)

with parameters: B1 = 0.691663, B2 = 0.4079426, B3 = 0.8974794,  $\lambda 1 = 0.0684043 \,\mu m$ ,  $\lambda 2 = 0.1162414$ , and  $\lambda 3 =$ 9.896161, where  $\lambda j = 2\pi c/\omega j$  and c is the speed of light in vacuum [15]. The optical fiber core refractive index for the simulations was obtained, according to the specifications from the fiber manufacturer, by increasing the refractive index of the cladding 0.36%.

## 130 D. Degrees Brix (°Bx) Sensor Monitored by Both Wavelength 131 and Phase Shift Detection

Typically, sensors are characterized by tracking the wave-132 length of an attenuation band using an optical spectrum an-133 alyzer, or alternatively by measuring the intensity variations 134 at a fixed wavelength using a power meter. The fast Fourier 135 transform (FFT) analysis, which permits to extract the phase of 136 the optical spectrum, is not a broadly used technique despite 137 it provides useful and clear information to be used in sensing 138 applications and permits to use interrogators instead of optical 139 spectrum analyzers [16], [17]. 140

The sinusoidal spectrum of the SMS sensors after etching permits to see a sharp peak corresponding to the fundamental frequency (see in Fig. 4 the comparison between the magnitude of the fast Fourier transform before and after etching). Consequently, it is possible to obtain a phase sensitive device by tracking the phase of this fundamental frequency as a function of the parameter to detect.

In order to probe the feasibility of this method for the developed sensor shown here, a degrees Brix sensor by phase shift detection is presented. The ITO thickness film to the sensor used in this application is approximately 60 nm. A MATLAB



Fig. 5. Evolution of the wavelength spectrum due to reduction of the diameter by HF etching: (a) Experimental; (b) theoretical.

script was implemented to obtain the phase of the fundamental 152 frequency in the optical spectrum response of the sensor. 153

Degrees Brix is a scale of relative density used in the sugar and winemaking industry. It indicates the percentage of cane sugar by weight in a solution or juice of unfermented grapes. Its measurement is crucial in many applications, such as fruit juice, carbonated beverage industry, and wine making.

The solutions used here were prepared by dissolving sucrose 159 in distilled water. One (1) °Bx equals one (1) gram of sucrose 160 dissolved in 100 grams of solution. 161

#### E. Phase Shift and Temperature Cross Sensitivity

A 40-nm thickness ITO thin-film deposited sensor fabricated 163 following the same procedure described for Sensor 2 was placed 164 in a water cell with temperature control. The temperature was 165 set to 40 °C and the spectra started being recorded after reaching 166 this set point, for 10 minutes. After this time, the control was set 167 to 30 °C and kept at this constant temperature for 20 minutes. 168 Finally, the temperature control was set back at 40 °C for 10 169 minutes once this temperature was reached. The real time phase 170 shift was recorded and processed along with the entire procedure 171 of temperature variation. 172

## A. Diameter Reduction

The experimental and theoretical evolution of the wavelength 175 spectrum as a function of the fiber diameter are depicted in 176 Fig. 5(a) and (b) respectively. 177

Fig. 6(a) and (b) show the theoretical and experimental transmission spectra for both, etched and unetched fibers. Video files 179 of this experimental and theoretical evolution can be found in 180 the supplementary material of this manuscript. According to [9], 181 [18], the diameter reduction is proportional to the sensitivity increase to refractive index. Consequently, a reduction from 125 to 25  $\mu$ m should lead to a fivefold increase. 184



Fig. 6. Comparison between the spectra of unetched and etched fibers: (a) Experimental; (b) theoretical.



Fig. 7. Experimental evolution of the spectra during ITO deposition from: (a) Sensor 2; (b) Sensor 3.

In addition to the structural integrity of the fiber, it was observed that at this diameter the transmission evolved into a quasisinusoidal spectrum for a length of the coreless MMF of 14 mm.
This behavior facilitates both the presence of multiple attenuation bands and the shift phase study.

## 190 B. Film Deposition

Fig. 7(a) and (b) present the spectral response obtained fromSensors 2 and 3 during the ITO films deposition.

For both cases, the transmission spectra underwent redshifts during the deposition, proving the existence of a relationship between the spectral position and the ITO film thickness. Sensor 2 and Sensor 3 exhibited 23 nm and 40 nm redshifts, respectively. Fig. 8(a) and (b), and Fig. 9(a) and (b) show the experimental and theoretical spectra before and after the ITO film deposition for Sensors 2 and 3, respectively.

It can be observed that the experimental redshifts due to the ITO deposition were in essence the same as those obtained theo-



Fig. 8. Initial and final transmission spectra due to the ITO thin-film deposition on Sensor 2: (a) Experimental; (b) theoretical.



Fig. 9. Initial and final transmission spectra due to the ITO thin film deposition on Sensor 3: (a) Experimental; (b) theoretical.

retically. This probes that the transmission spectrum experiences 202 a redshift as thin-film thickness grows. 203

Based on theoretical analysis, it can be concluded that the 204 thicknesses of the deposited thin-film were of 40 nm and 60 205 nm to Sensor 2 and Sensor 3, respectively. To support the good 206 match between the experimental and theoretical results, Sensor 2 207 was cleaved and observed using a scanning electron microscope 208 (SEM). Fig. 10 shows the cross section, where the measure-209 ment of the film thickness was 44.18 nm. This supposes a 10% 210 deviation with respect to the theoretical value for this sensor. 211

#### C. Wavelength Shift Characterization of the Device 212

Figs. 11, 12, and 13 show the theoretical and experimental 213 transmission spectra as a function of wavelength for various 214 refractive indices. A redshift can be observed in all cases when 215 the RI increases, and there is a good agreement between the 216 theoretical and experimental results. 217

Fig. 14 illustrates the wavelength shift as a function of the 218 external refractive index, which allows the sensitivity to be calculated both experimentally [see Fig. 14(a)] and theoretically 220 [see Fig. 14(b)]. In both cases the wavelength position was 221 taken with the attenuation band closer to 1550 nm. 222



Fig. 10. SEM images of Sensor 2: Diameter 25  $\mu$ m and ITO thin film 44 nm.



Fig. 11. Transmission spectrum for Sensor 1 as a function of the wavelength for different surrounding media refractive indices: (a) Experimental; (b) theoretical.

It can be noticed that an increase in the thin-film thickness leads to a higher sensitivity. Table I shows a summary of the sensitivities in the 1.32–1.35 refractive index range. The experimental sensitivity improves that obtained in [9]. Indeed, even though a sensitivity of 1200 nm/RIU was attained in that work, the SRI range was 1.32–1.38, with a higher sensitivity. More-



Fig. 12. Transmission spectrum for Sensor 2 as a function of the wavelength for different surrounding media refractive indices: a) Experimental; b) theoretical.



Fig. 13. Transmission a) experimental and b) theoretical for Sensor 3 as a function of the wavelength for different surrounding media refractive indices.



Fig. 14. Wavelength shift with refractive index to Sensor 1, 2 and 3, for both, a) experimental and b) theoretical cases.

TABLE I Comparison Sensitivities Obtain Experimentally and Theoretically to A 1.32–1.35 RIU Range

Thin-film Thickness (nm)	Experimental Sensitivity	Theoretical Sensitivity
Uncoated	335 nm/RIU	454  nm/RIU
		15 1 1111/1010
	$R^2 = 0.9812$	R <sup>2</sup> =0.9669
$\sim 40$	1062 nm/RIU	1131 nm/RIU
	R <sup>2</sup> =0.9906	R <sup>2</sup> =0.999
$\sim 60$	1442 nm/RIU R <sup>2</sup> =0.953	1536 nm/RIU R <sup>2</sup> =0.9951



Fig. 15. Transmission as a function of the wavelength to different ° Bx of an (a) etched SMS configuration without thin-film and (b) a 60 nm thin-film Thickness ITO etched SMS configuration.

over, though overcome by LPFGs optimized with a hard etching
[19], the device presented here is comparable with the sensitivity
obtained with LPFGs optimized with a soft etching [20], which
along with the possibility to monitor the phase shift indicates
that it is an interesting device for biosensing applications, where
a high degree of accuracy.

# D. Degrees Brix (°Bx) Sensor Monitored by Both Wavelength and Phase Shift Detection

Fig. 15 shows transmission as a function of the wavelength for different solutions of sucrose in water addressing two cases: an etched SMS configuration without thin-film and a 60 nm thickness ITO thin-film deposited on it. Both cases show a redshift when the probe was immersed in.

The magnitude of the wavelength shift is more notorious in the coated fiber, regardless of the attenuation observed, according to what has been observed in the previous section.

Fig. 16(a) and (b) present the wavelength shift of an attenuation band and the phase shift for both the etched SMS configuration without ITO coating and the same configuration type with a 60 nm thickness ITO thin-film. A simple linear fit of the results evidences that the ITO film permitted to obtain a three-fold sensitivity increase in both cases.



Fig. 16. Phase shift of a brix grades sensor: (a) Without ITO thin-film; (b) with 60 nm thickness ITO thin-film.



Fig. 17. (a) Wavelength shift and (b) phase shift temperature cross sensitivity.

#### E. Temperature Cross Sensitivity

Fig. 17(a) and (b) show the behavior of the wavelength shift 252 of an attenuation band and the phase shift with the temperature 253 setting as described in Section II-E. This probe was realized 254 with the 60 nm thin-film thickness sensor. The phase behavior 255 observed followed a trend imposed by the temperature's profile 256 generated as a consequence of the set points established. The 257 sensitivity was 0.3 nm/°C and 0.034 rad/°C. 258

A 0.14 °Bx/°C of sensitivity was calculated for the ITO thinfilm coated sensors analyzed in the previous section. 260

This manuscript presented the optimization of SMS fiber 262 structures with a combined application of two techniques: etching and deposition of a thin-film. By an adequate design it 264 was possible to track both wavelength shifts of the optical spectrum and, by applying a simple FFT measurement technique, the 266

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used in the analysis is an easy method that can be most applicable 268 to networks that require narrow band, multiplexing capability 269 270 and that have some problems related with high losses and noise. The results also showed that the sensitivity obtained for this 271 configuration of SMS was enhanced by reduction of the fiber 272 diameter and by increasing the ITO film thickness. A good 273 agreement was achieved between the experimental and the sim-274 ulated approaches for this sensing device. A sensitivity of 1442 275

phase shift of the fundamental frequency. The FFT measurement

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nm/RIU was obtained by tracking the wavelength shift in a SMS with 25  $\mu$ m diameter and a 60 nm ITO thickness film, whereas for the same device, the FFT phase shift analysis showed a 0.24 rad/RIU sensitivity.

These sensitivities, which are in the order of magnitude of 280 other structures such as long period fiber gratings (LPFGs) (but 281 with an inherently simpler manufacture process), place the de-282 veloped sensing device as a good option for applications where 283 high sensitivities and compact structures are required. As an 284 example, a degrees Brix sensor has been presented, where the 285 deposition of an ITO thin-film enhances the sensitivity of the 286 device by a factor of 3. 287

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