# Lossy Mode Resonance optical sensors based on indium-gallium-zinc oxide thin film

A. Ozcariz[1,*], M. Dominik[2], M. Smietana[2], C. R. Zamarreño[1], I. Del Villar[1], F. J. Arregui[1]

[1]Department of Electrical and Electronical Engineering, Public University of Navarre, 31006 Pamplona, Spain
[2]Institute of Microelectronics and Optoelectronics, Warsaw University of Technology, Koszykowa 75, 00-662 Warszawa, Poland
*e-mail: aritz.ozcariz@unavarra.es

**Abstract:** This work discusses an application of electrically conductive and optically transparent indium-gallium-zinc oxide (IGZO) thin films for fabrication of lossy mode resonance (LMR) based optical fiber refractometers. The films have been deposited on both 200 µm in diameter multimode fused silica fiber and D-shaped fiber structure. A sensitivity of 12,929 nm/RIU was obtained in the refractive index range between 1.39 and 1.42. In case of D-shaped fiber structure the effect of polishing depth on the LMR phenomenon has also been studied.

## Keywords

Optical fiber; Refractometer; Optical Sensor; D-Shape Optical Fiber; Lossy Mode Resonances (LMR); Indium-Gallium-Zinc Oxide (IGZO)

## Introduction

Optical fiber sensors based on lossy mode resonance (LMR) have already been successfully applied in recent years for measuring different parameters [1–3]. For LMR generation these sensors require a thin film deposited on an optical waveguide, such as a core of an optical fiber. In order to be able to generate LMR, the real part of the film's permittivity must be positive and higher in magnitude than both the imaginary part of the film's permittivity and the real part of the surrounding medium's permittivity [3,4]. At these conditions, there is a mode guided in the optical fiber that at certain wavelength experiences a transition to guiding with losses in the thin film. This phenomenon induces an increase in the imaginary part of the effective index of the rest of modes guided in the core of the optical fiber. As a result, an attenuation at a specific wavelength range is observed, which is called LMR. The guiding conditions of the lossy mode are dependent on the refractive index of the medium surrounding the coated fiber which corresponds to its permittivity, thus the refractive index change induces a shift in the LMR wavelength. Therefore, this phenomenon may be applied when fabrication of a refractometer is considered. Tuning of the thin film properties by its fabrication parameters permits to obtain the best performance of the sensor in terms of sensitivity [5]. Therefore, studies on material used for the sensor fabrication is of high importance. Several materials have already been used for obtaining LMR-based sensors, that include polymers [6], oxides of various metals, i.e., tin [5,7], indium tin (ITO) [8–11], zinc [12,13], hafnium and tantalum [14] or titanium [15], as well as silicon nitride [16], and diamond-like carbon [17,18]. The obtained devices have been used in a broad range of applications that include e.g., determination of pH or humidity, detection of gases (hydrogen, ammonia) or p-cresol.

The expected optimal properties of the thin film required for LMR generation have already been discussed in the literature [4,19]. In general the real part of the electrical permittivity must be positive and higher in magnitude than both its imaginary part, and the real part of the

permittivity of the external medium. However, there are other thin film properties that must be taken into consideration. In particular, the film thickness plays an important role. Thus an application of deposition technique that facilitates a precise control over thin film parameter is highly expected. Moreover, some materials may change their properties in temperature, humidity, or presence of certain gases, what defines their application. That is why it is required to study various materials in order to extend scope and possibilities of the LMR sensors application in different fields.

In this work we focus on the indium gallium zinc oxide (IGZO) as LMR-supporting thin film material. Zinc oxides have already been widely studied for fabrication of various sensors [20–22], also when doped with other materials such as aluminum (AZO) [23], indium [24] and gallium [25]. IGZO has been extensively used as a transparent electrode material in liquid crystal displays [26], as well as an active layer in mainly transistor-based ozone [25], salinity [27], temperature [28] or glucose [29] sensors. IGZO fulfills the optical properties described above for LMR generation and also adds some benefits such as its stability [26,30,31] in comparison with other materials such as ITO. The fact that it possesses a high electron mobility makes it a great candidate for gas sensing applications [32]. Here, we show experimental results for IGZO-coated optical fiber LMR sensors based on two different kinds of fibers, i.e., a multimode fiber with exposed core and a D-shape fiber. Differences observed for each of the configurations are discussed. While multimode fibers allow to fabricate simpler and low-cost sensors for wider spectral range, D-shape fibers facilitate the use of polarized light and the separation of transversal electric (TE) and transversal magnetic (TM) modes. This is important because the LMR takes place for both modes, but the resonances are observed at slightly different wavelength and offer different sensitivities [8]. In addition, the isolation of TE and TM modes permits to obtain better spectrally defined resonances and thus offer higher measurement resolution. For the IZGO-coated D-shaped fiber we also studied the effect of polishing depth on the LMR effect.

A functionalized layer can be fabricated on top of the LMR supporting film. If refractive index of this layer is sensitive to the presence of certain element, this can be observed as a shift in the LMR. In this way, the development of LMR-based refractometers is the basis for the fabrication of several kinds of sensors. For example, a sensor has been reported consisting on a ITO coated D-Shape fiber, which has been coated with PAH/PSS bilayers, ending with a CRP-aptamer coating [33]. This device showed a shift in the LMR when CRP (C-Reactive Protein) binds to the CRP aptamers in the fiber. A similar approach using SnO2 as LMR supporting material obtained a femtomolar concentration detection in human serum [34].

## Experimental details

Two kind of fibers were coated in this experiment: a multimode FT200EMT plastic-clad silica with 200/225 µm core/cladding diameter from Thorlabs Inc. with 4 cm segment of an exposed core and D-shape optical fibers from Phoenix Photonics. These fibers were coated in a DC sputtering chamber (K675XD from Quorum Technologies Ltd.), using a 57 mm diameter IGZO sputtering target from Beijing Loyaltarget Technology Co. The sputtering was performed under a pressure of $8 \cdot 10^{-2}$ mbar and at 100 mA current. The thickness of the coating was controlled by the deposition time and measured using a field emission scanning electron microscope (SEM) UltraPlus from Carl Zeiss Inc. with an in-lens detector at 3 kV and an aperture diameter of 30 µm. Spectroscopic ellipsometry measurements were performed using an UVISEL 2 ellipsometer (Horiba) to obtain the dispersion curves of the film. During the deposition, the multimode fibers

were connected to a halogen light source (Spectral Products Inc.) and a couple of spectrometers (USB2000+XR1 and NIR512 from Ocean Optics Inc.) through a bifurcated fiber (Ocean Optics Inc.). The setup shown in Fig. 1A allowed to observe the evolution of the resonances with the coating thickness and determine the suitable deposition time for the resonances present at the desired spectral range. A similar procedure was carried out using the D-shape fiber and a superluminescent emitting diode light source (HP83437A) and an Optical Spectrum Analyzer (HP86142A) were used, as well as a linear polarizer (Phoenix Photonics) and a polarization controller (Thorlabs FPC032). The applied setup is shown in Fig. 1B. The use of standard single mode fiber allows the drift in the polarization due to small birefringence in the glass but, it should be noted that for the current experiment the polarization state must be maintained only between the polarizer and the D-Shaped segment. This is possible keeping this short length of the fiber firmly fixed to observe the separation of modes.

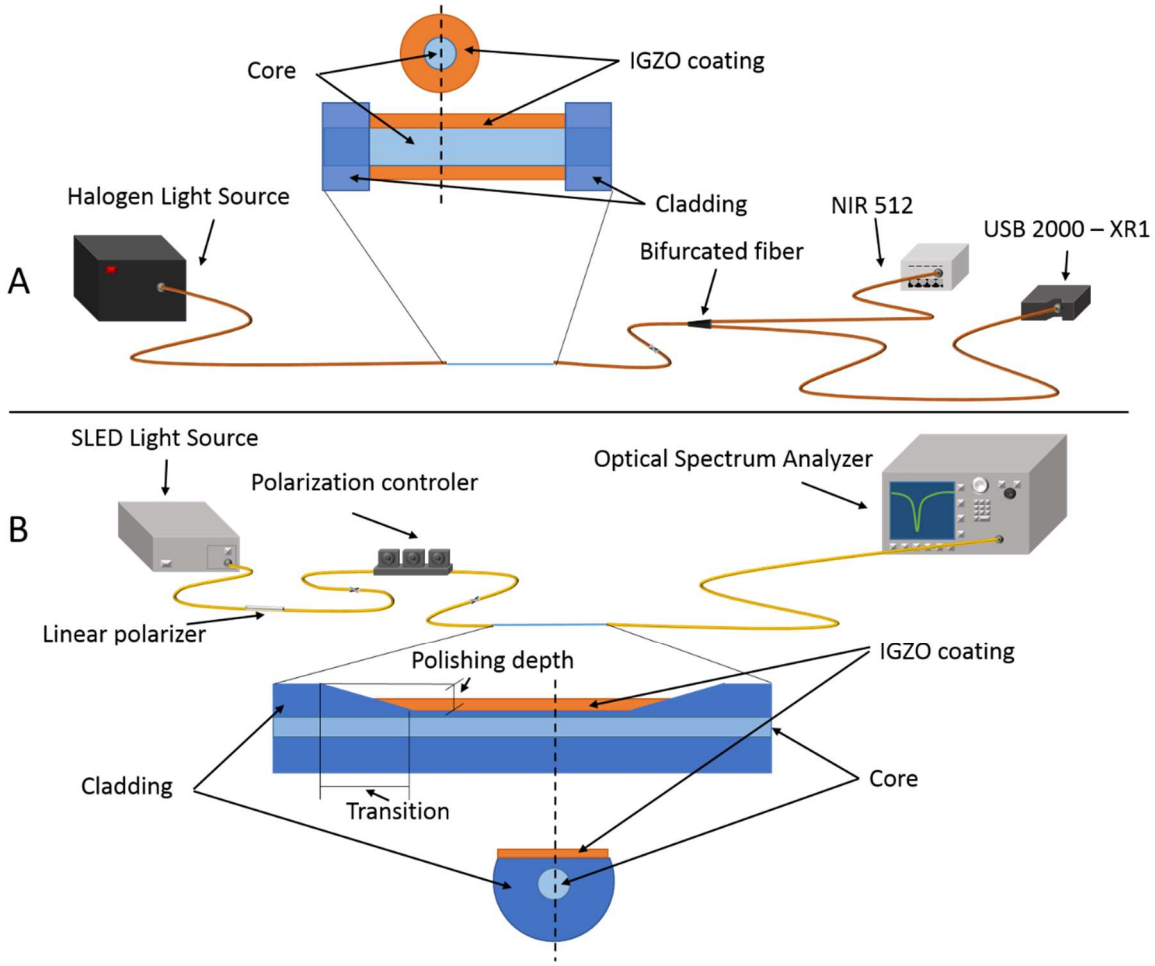


Fig. 1. Setups used for investigations of devices based on (A) multimode and (B) D-shape fiber. A detail of the sensitive region and a cross-sectional view are also shown for both the cases. In addition, the D-Shape fiber in (B) also includes a schematic representation of the transition zone between the regular fiber and the uniformly polished zone.

## Results

The IGZO coating thickness was studied first. The growth rate was determined by scanning electron microscope (SEM) measurements of a reference silicon wafer cross-section. The wafer was located next to the fiber sample in the sputtering chamber and the coating thickness was

as shown in Fig. 2A. This process was repeated for 5 times in order to study the deposition method. For the applied deposition conditions growth rate reached 21.3 nm/min. In addition, energy-dispersive X-Ray spectroscopy (EDX) was performed to verify composition of the IGZO coating, in particular presence of indium, gallium and zinc (Fig. 2B).

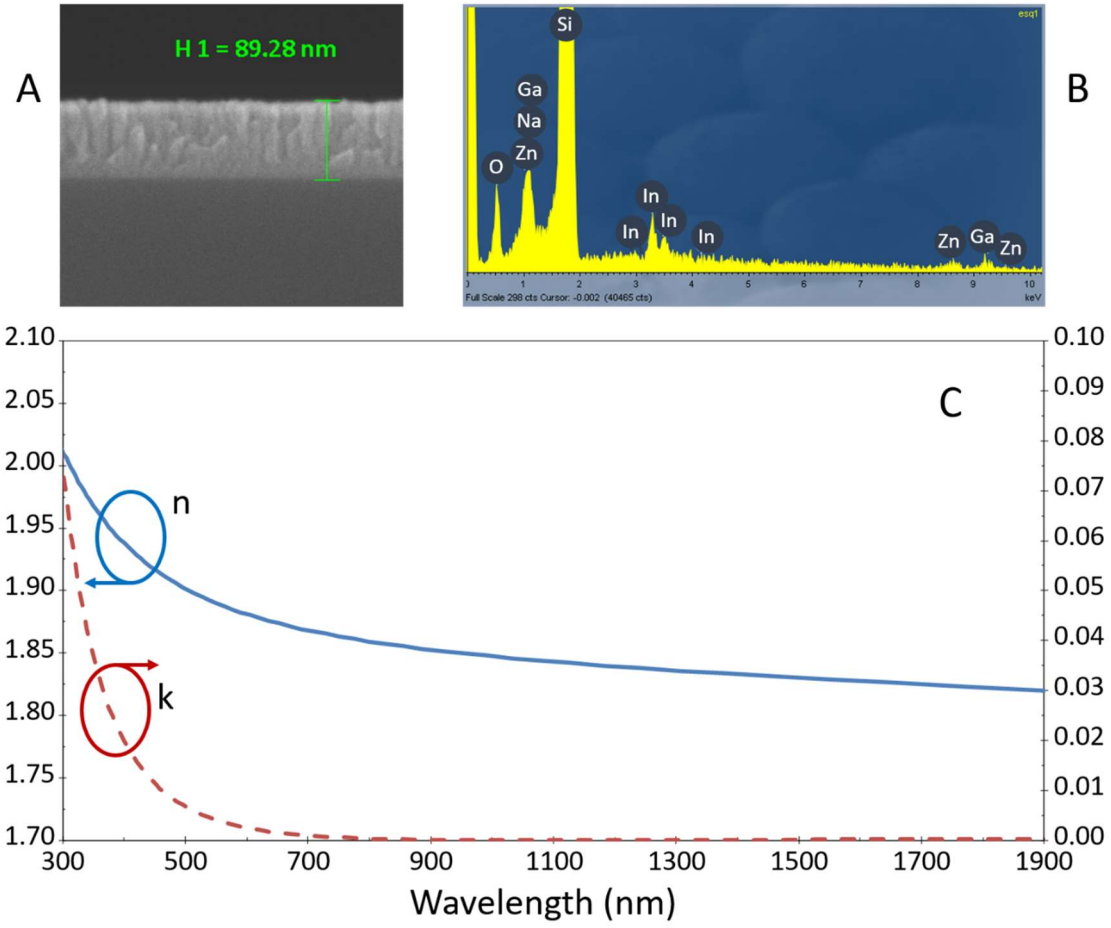


Fig. 2. (A) SEM image and (B) EDX spectrum of the IGZO thin film deposited on a reference silicon wafer. The presence of indium, gallium and zinc is confirmed, as well as oxygen and silicon substrate. (C) Dispersion curves of IGZO obtained from spectroscopic ellipsometry.

The spectroscopic ellipsometry measurement (Fig. 2C) shows that IGZO fulfils the requirements for LMR generation, as the refractive index (n) is relatively high and the extinction coefficient (k) is close to zero, approx. 1.82 and 0.0001 respectively in the NIR range (1550 nm).

In Fig. 3 can be observed shift of the resonances towards higher wavelengths with the increase in IGZO thickness. Up to 7 LMRs can be distinguished after a 20 minutes long deposition, what corresponds to 426 nm of thickness, according to the determined growth rate. Note that for the 1$^{st}$ order LMR two diverging components can be distinguished that correspond to the TE and TM modes. These components are not observed for the higher order resonances as they are closer in wavelength. Another factor taken into consideration is that each resonance has different sensitivity to changes in IGZO thickness. Each higher resonance offer slightly lower sensitivity than the resonance of lower order. The 1$^{st}$ order resonance is the most sensitive one. In addition, according to [10] this dependence is similar for the sensitivity to surrounding refractive index (SRI) - the 1$^{st}$ order LMR is the most sensitive one.

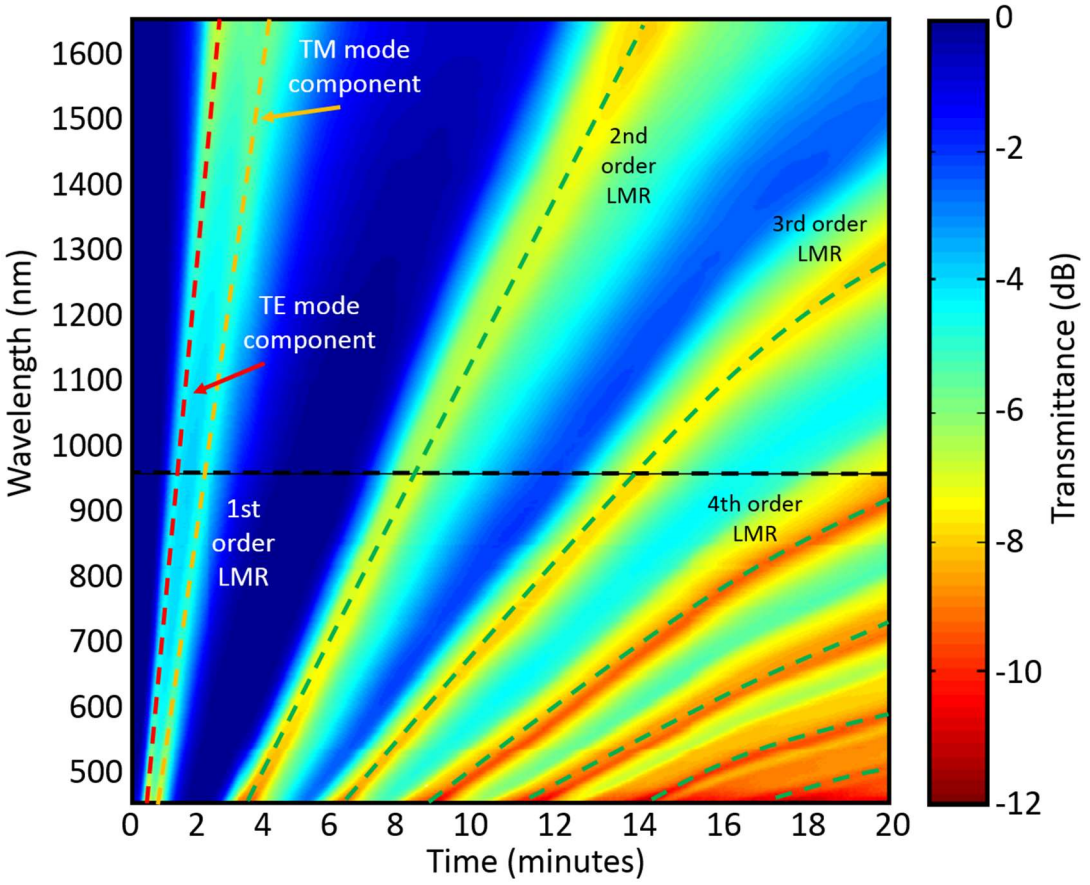


Fig. 3. Evolution of the multimode fiber transmission spectrum with IGZO deposition time. The increase in IGZO thickness induces shift of the resonances to longer wavelength. Up to 7 resonances can be observed. For the 1st LMR TE and TM modes can be differentiated.

The two components corresponding to the TE and TM modes result in an undesired effect for the multimode optical fiber (with no polarizer) - a resonance broadening, that hinders the observation of each mode separately and allows for observation of an envelope only. On the other hand, the 2nd order LMR, although a bit less sensitive, is narrower and enables a better observation and determination of its resonance wavelength. Therefore, a deposition process was performed on the multimode optical fiber targeting on tuning the 2nd order resonance to be observed in the VIS range (thickness of 106 nm). Once coated, the fiber was stored for 24 h. at room temperature (23 ºC) to allow the thin film to stabilize. It is known that a small oxygen absorption typically observed for fresh sputtered thin films, may change the optical properties of the film and induce a shift of the LMR. Next, it was immersed in water/glycerin solutions to increase SRI and observe the shift of the LMR. A red-shift is induced with the SRI (Fig. 4A).

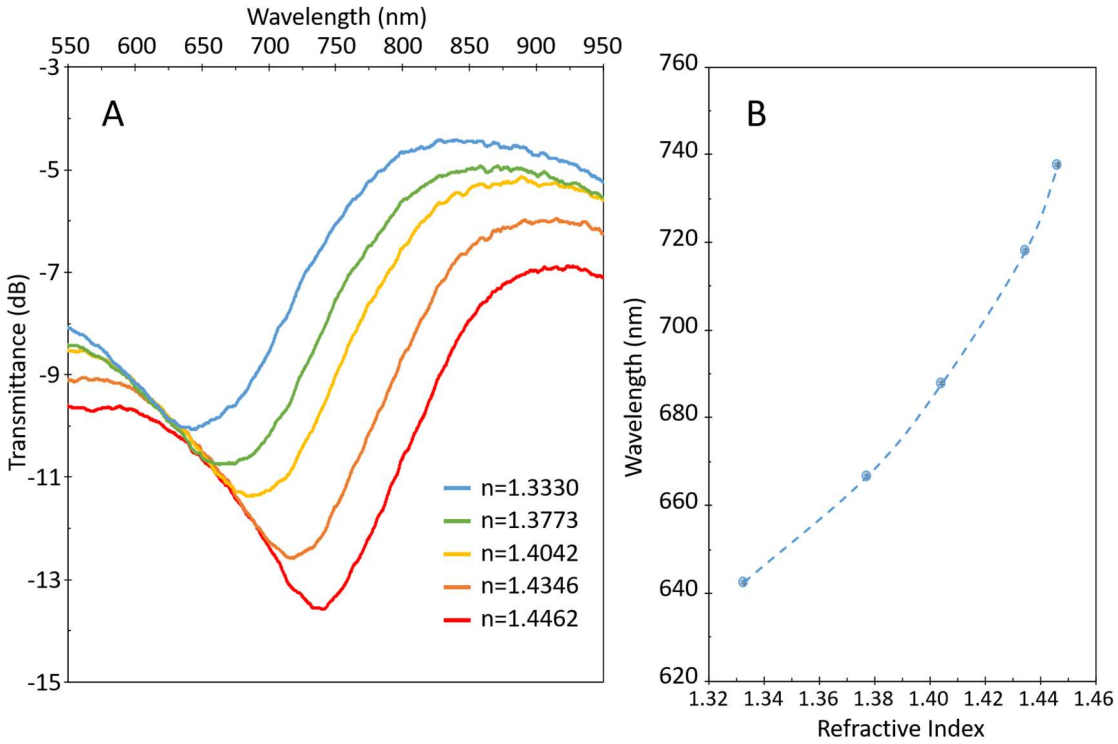


Fig. 4. (A) Transmission spectra of the multimode fiber-based sensor with coated with 106 nm IGZO coating when it is submerged in various SRI. (B) Transmission minimum dependence on SRI. The sensitivity increases with SRI. Note that the error in wavelength is ±0.27 nm and ±0.0001 in RI.

The LMR wavelength has been obtained using a Matlab-based algorithm, which allowed to determine the wavelength corresponding to each SRI. The sensitivity is non-linear dependent on SRI and wavelength and therefore, we expect an increase in sensitivity for concentrations of higher RI [19]. In our case, it can reach 1666 nm/RIU (Fig. 4B). This value is close to that obtained using ITO as a thin film material [35].

A similar procedure was applied to D-shape optical fibers. In this case, two different fibers were coated, i.e., with 4 dB and 1 dB attenuation measured during polishing at $\lambda$=1330 nm and for SRI of 1.5. The 4 dB fiber structure underwent a deeper polishing making the distance between the core and coating shorter. In order to compare the influence of polishing depth, the IGZO coatings on both the fibers were tuned to show the LMR at the same spectral window and for similar SRI. These coatings were approximately 85 nm in thickness.

There are significant differences between performance of both the fiber structures. For the 4 dB structure it can be seen the first LMR composed by TE (Fig. 5A) and TM (Fig. 5B) modes. In both the cases a maximum attenuation of up to 30-35 dB was achieved. However, a great distortion is present with numerous side-lobes that make difficult to precisely determine the attenuation peak. These resonances achieve an average sensitivity (Fig. 6) of 5,111 nm/RIU and 12,929 nm/RIU for TE and TM, respectively. This difference in sensitivity is due to the fact that higher values of SRI were investigated for the TM mode. As it has been reported, the sensitivity of the LMR-based sensors increases with both the SRI and the wavelength range [19].

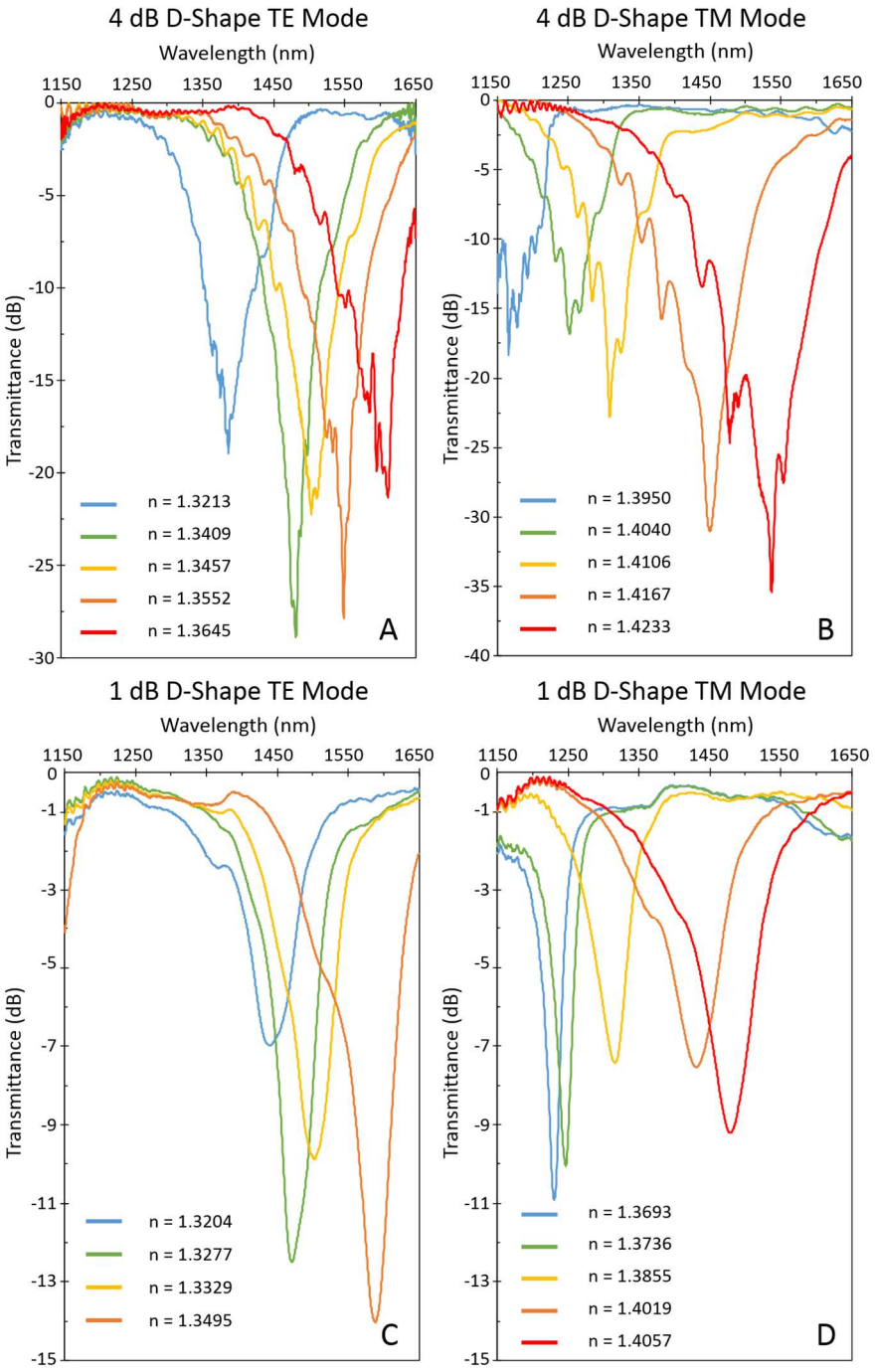


Fig. 5. Transmission spectra of the D-shape fibers. In (A) and (B) are shown the LMRs generated for the TE and TM modes and the 4 dB structure. In (C) and (D) in turn are shown the resonances for the TE and TM modes and the 1 dB structure.

To study the effect of the polishing depth on the LMR effect, it is interesting to compare the results obtained using the 4 dB (Fig. 5A and 5B) with those obtained using the 1 dB D-shape fiber structure (Fig. 5C and 5D). The LMRs of the 1 dB D-shape fiber are lower in transmittance by approx. 14 dB and better defined shape with no side lobes. For these resonances a direct determination of the resonance wavelength is possible and they offer an average sensitivity of 5,062 nm/RIU and 6,855 nm/RIU for the $LMR_{TE}$ and $LMR_{TM}$, respectively. Comparing again these results with those obtained using ITO in this configuration [8], we can observe that the sensitivity obtained here is also similar for the same SRI range. Table 1 shows a comparison of the

sensitivities obtained using different materials. The difference in amplitude of these resonances has a direct effect on their FWHM (full width at half maximum). This parameter, along with the figure of merit (FOM), obtained dividing the sensitivity by the FWHM, is often used to compare the performance of various devices, including refractometers. The use of D-shape fiber and polarized light permits to obtain optimal values of these parameters. The parameters have not been described for other IGZO-based LMR sensors. The resonances observed in the 1 dB fiber have an average FWHM of 48 and 44.8 nm for the TE and TM modes respectively and their FOM values are 105.46 and 155.09 $RIU^{-1}$. For the 4 dB fiber the parameters are even better and reach an average FWHM of 12.9 and 11.1 nm and FOM reaching 396.20 and 1164.77 $RIU^{-1}$, for the $LMR_{TE}$ and $LMR_{TM}$ respectively, although this resonances present the mentioned distortion.

Table 1: A brief comparison of the sensitivity obtained for LMR-based optical sensors fabricated with different coating materials.

| Material | Fiber Configuration | Spectrum Range (nm) | R.I. Range | Sensitivity (nm/RIU) | References |
|---|---|---|---|---|---|
| **IGZO** | Multimode | 600-900 | 1.32-1.45 | 1,666 | Current work |
| **IGZO** | D-Shape | 1100-1600 | 1.39-1.43 | 12,929 | Current work |
| **ZnO** | Multimode | 300-450 | 1.32-1.44 | 760 | [12] |
| **ZnO+NRs** | Multimode | 300-450 | 1.32-1.44 | 1,160 | [12] |
| **ZrO2** | Multimode | 600-900 | 1.41-1.43 | 880 | [14] |
| **SixNy** | Multimode | 600-900 | 1.43-1.45 | 593.5 | [14] |
| **ITO** | Multimode | 800-1600 | 1.32-1.44 | 1,520 | [35] |
| | D-Shape | 1100-1600 | 1.365-1.38 | 8,742 | [8] |
| | D-Shape | 1100-1600 | 1.447-1.449 | 304,361 | [9] |
| **InO2** | Multimode | 1100-1600 | 1.32-1.39 | 4,000 | [35] |
| **SnO2** | Multimode | 1100-1600 | 1.333-1.42 | 5,390 | [7] |
| | D-Shape | 1100-1600 | 1.32-1.326 | 14,501 | [9] |
| | D-Shape | 1100-1600 | 1.448-1.449 | 1,087,889 | [5] |

While the sensitivity for the TE mode is similar in both 1 dB and 4 dB cases, the sensitivity for the TM mode in case of 1 dB structure seems considerably lower. However, it must be taken into account, that in this experiment the $LMR_{TM}$ for the 1 dB structure appears at slightly shorter wavelength range and at lower SRI than for the 4 dB structure. That is why the obtained sensitivity for 1 dB structure is also lower than for the 4 dB one. When the sensitivity obtained for both the structures in a similar SRI range is compared (1.38-1.41), the sensitivity values for both fibers are similar and reach 8,120 nm/RIU and 8,974 nm/RIU for the 1 dB and 4 dB structures, respectively.

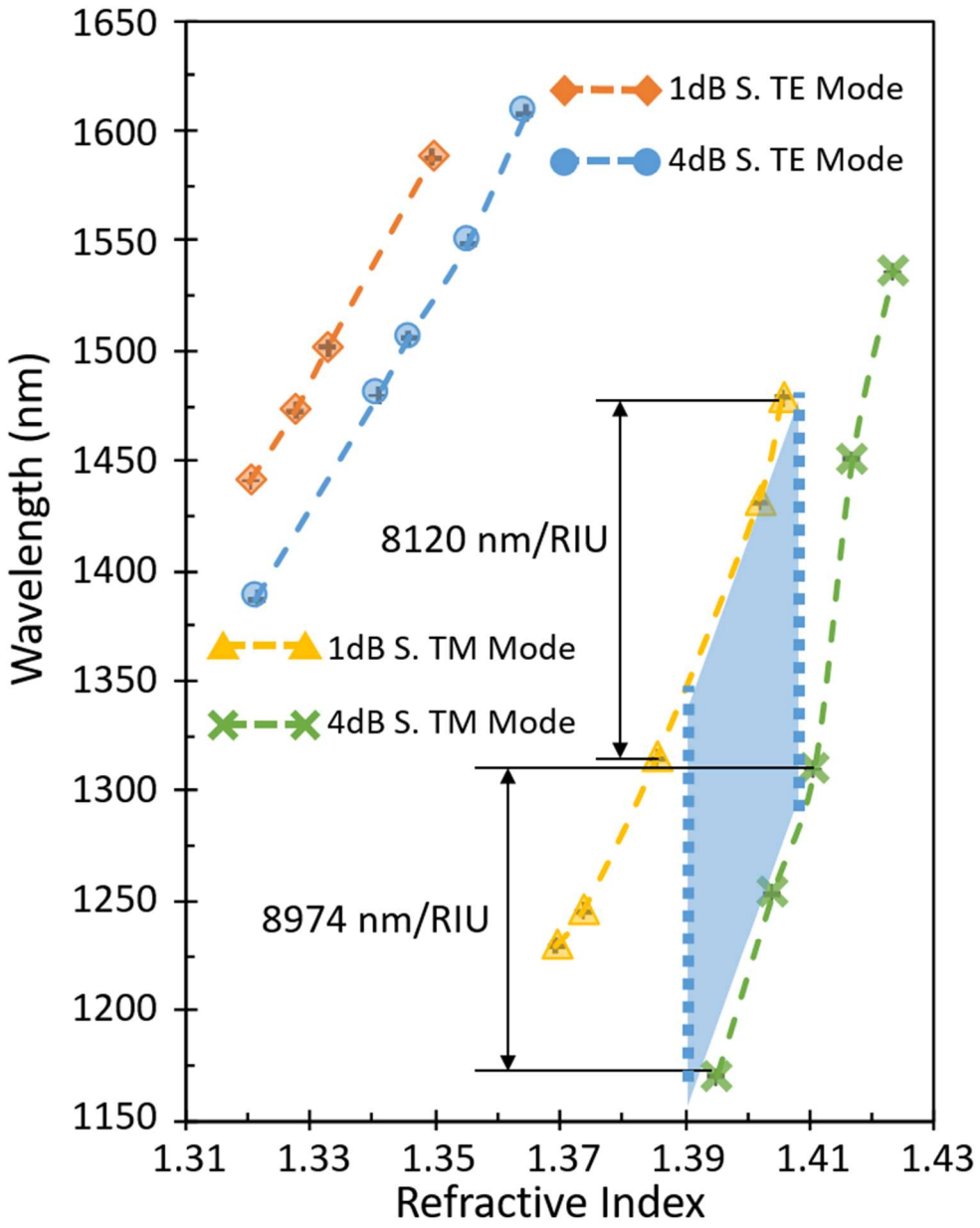


Fig. 6. LMR wavelength of the resonances generated for both the TE and TM modes when submerged in several SRI solutions. It can be observed that the sensitivity is similar for both the structures in the same SRI range, despite the LMR are slightly shifted in wavelength. Note the error is 0.5 nm for wavelength and 0.0001 for RI.

We can conclude that the main difference in the use of D-shape fibers with different polishing degrees is the transmittance at the resonances and the smoothness of the response. The difference in the transmittance can be explained by the polishing depth, i.e., a deeper polishing induces an increase in the evanescent field. Consequently, the resonance depth is higher. However, the shape of the resonances and the generation of a ripple effect is a more complex phenomenon. In earlier works [19], it has been proven how the variation of the imaginary part of the refractive index (k) in the coating can affect the shape of the LMR. However, since the deposition has been performed in the same conditions and using the same materials, we can assume there is no variation in the optical properties of both coatings. The main difference between both cases is originated by the geometry of the fibers. As a result of fabrication process,

we can expect formation of a transition zone between the regular single-mode fiber and the D-shape segment. This transition zone is difficult to study since it depends on the fabrication process and the degree of polishing. However, it can be considered that the deeper the polishing is, the longer and uneven is the transition zone. Therefore, the effect of this zone on the resonance should be stronger when there is a deeper polishing (as for the 4 dB D-shape fiber). We suggest that the transition zone could be responsible for excitation of certain other modes that generate ripples and distortions in the LMR spectrum.

As previously mentioned, these refractometers are the basis for the fabrication of chemical sensors. Comparing the RI sensitivity achieved using IGZO thin film with that achieved in LMR based sensors using different materials, we can estimate the potential sensitivity that could achieve in chemical sensors. For instance, a LMR based multimode fiber salivary cortisol sensor using ZnO thin film and MIP layer had been previously developed, obtaining a sensitivity of 12.86 nm/log(g/ml) [36]. Since the RI sensitivity obtained by our multimode fiber IGZO sensor is 2.19 times higher than that of similar ZnO based LMR sensor (760 nm/RIU [12]), we could estimate that using IGZO the cortisol sensitivity could reach 28.19 nm/log(g/ml), which could rise up to 218.77 nm/log(g/ml) using a D-Shape fiber setup since it achieves a sensitivity 17.01 times higher. Similarly, a previously reported paper also demonstrated de use of ZnO based LMR sensor for urinary p-cresol detection [13], obtaining a sensitivity of 11.86 nm/μM, which could be improved up to 201.76 nm/μM using an IGZO coated D-Shape fiber.

## Conclusions

Several optical fiber sensors have been fabricated using IGZO coating as a LMR-supporting material. The presence of LMR for several mode orders have been demonstrated and the dependence of the LMRs on the IGZO coating has been studied. A sensitivity of 1,666 nm/RIU has been obtained using a multimode fiber setup.

For D-shape fiber sensors in turn a maximum sensitivity of 12,929 nm/RIU has been reached. It has been demonstrated that the polishing depth has no significant impact on the sensitivity, but it affects transmittance and shape of the resonances. The use of a less polished fiber allows for achieving resonances with higher transmittance, but their shape and the resonance wavelength are better defined.

Finally, as it is mentioned above, the use of IGZO as supporting material for LMR-based optical fiber sensors is especially justified by properties of the IGZO thin film, that may allow for fabrication of more stable sensors in comparison to ITO or AZO. Besides, the high electron mobility of this material enhances its interaction with gases making it also promising for gas sensing, also in biomedical applications.

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