Turn Around Point Long Period Fiber Gratings With Coupling to Asymmetric Cladding Modes Fabricated by a Femtosecond Laser and Coated With Titanium Dioxide

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Abstract—A detailed study of turn around point (TAP) long period fiber gratings (LPFGs) with coupling to the asymmetric cladding modes of a standard single-mode fiber (SMF-28e), fabricated by femtosecond (fs) laser direct writing was realized. The entire fabrication process, including the coating with different titanium dioxide (TiO₂) film thicknesses of LPFGs and the corrections needed to achieve coated devices operating precisely in the TAP condition with coupling to the asymmetric cladding modes, was addressed. The significant fabrication details are also given, such as inscription periods, shape and localization of the refractive index modifications across the core. The fabrication process described allows the optimization of the LPFGs sensitivity in regards to the surrounding refractive index (SRI). Optimization of the writing parameters to obtain gratings working at the TAP for two different media surrounding the fiber (water and air) was achieved. It was demonstrated that for a grating period of 191.8 μ m, the LP_{1,12} mode exhibits a TAP at 1442.7 nm in air, and for a period of

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192.5 μ m, the same mode exhibits a TAP at 1448.6 nm in water. The LPFGs operating at the TAP in air and water were coated with 10, 20, and 30 nm thin TiO₂ film thicknesses and the spectral behavior characterized. The wavelength sensitivity to the surrounding refractive index (SRI) was assessed in the range between 1.3700 to 1.4120, and a maximum sensitivity of ~8051.4 nm/RIU was measured for the 192.5 μ m LPFG coated with a 30 nm thick TiO₂ film.

Index Terms—Femtosecond laser direct writing, optical fiber sensors, refractive index sensing, titanium dioxide thin films, turn around point long period fiber gratings.

I. INTRODUCTION

ONG period fiber gratings (LPFGs) have been under continual investigation for at least forty years [1]. The fabrication of the LPFGs using several methods has been demonstrated, including exposure to UV and CO₂ laser radiation [2]-[5], electric arc discharge [6], laser ablation [7], mechanical pressure [8], and also femtosecond (fs) laser writing [9]-[11]. The LPFGs inscription in different optical fibers, such as photonic crystal fibers [12], standard single-mode optical fibers [11], polarization-maintaining fibers [13], and photosensitive fibers were also extensively explored [7], [13]. The LPFGs are induced by a periodic modulation of the refractive index (RI) along the longitudinal axis of an optical fiber with periods greater than 100 μ m [1], [7], [14]. This modulation allows the coupling of light from the LP₀₁ core-guided mode into forward-propagating cladding-guided modes (LP1,m, linearly polarized modes with azimuthal and radial integer indices l and m, respectively) at or near-resonant wavelengths. The efficient coupling between the core mode and the co-propagating cladding modes occurs under the phase-matching condition, where the resonant wavelength of the LPFG, λ_{res} , is determined by [1], [8]:

$$\lambda_{res} = \left(n_{core}^{eff} \left(\lambda \right) - n_{cl \ l,m}^{eff} \left(\lambda \right) \right) \Lambda \tag{1}$$

where n_{core}^{eff} and $n_{cl\,l,m}^{eff}$ are the effective RI of the propagating core mode and m^{th} cladding modes, respectively, at wavelength λ , and \wedge is the grating period. The resonance wavelength sensitivity of the LPFGs to external perturbations relies on the effective RIs and the grating period, underlying

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to the propagation conditions in the core and cladding modes. The resonance wavelength shift caused by the changes in the effective RIs and the grating period allows the measurement of several parameters such as temperature, strain, and refractive index [1], [3], [8]. Regarding the LPFGs based RI sensors, they can be used efficiently in biological, chemical, and biochemical sensing applications [15], [16], [17]. The RI measure is defined as a shift of the resonance wavelength per RI Unit (RIU) [15]. The fabrication of LPFGs with high sensitivity to the surrounding refractive index (SRI) has been explored [16], [18], [19]. The methodologies reported include LPFGs operating in the turning around point (TAP) region, evanescent field enhancement by reducing cladding diameter, and mode transition (MT) [20] achieved by coating the LPFGs with a thin layer of material whose RI is higher than that of the cladding [16], [18], [19]. The high RI thin film coatings promote the transition between cladding guided modes in overlay guided modes inducing a re-distribution of the cladding modes, enhancing the evanescent wave interaction with the surrounding medium [16], [18], [19]. In addition, a combination of these methods by selecting a period very close to the TAP and making the fine tunning to operate precisely in the TAP by reducing the cladding diameter or by coating with a thin layer of material could further improve the SRI sensitivity of the LPFGs sensors [16], [18], [19].

The fabrication of LPFGs operating at the TAP without the use of post-fabrication technics (i.e., chemical etching to fine-tune the LPFGs operation around TAP) has proven to be a challenge, corroborated by the few manuscripts reported so far [2], [3], [11], [16], [21]. In addition, the fabrication of LPFGs operating at the TAP of asymmetric cladding modes for SRI sensing has not been discussed in detail; nevertheless, to our knowledge, fabrication of LPFGs allowing the coupling to asymmetric cladding mode was only demonstrated in [6], [22], [23].

The SRI sensitivity of the LPFGs sensors could be further improved, selecting a high RI thin film, such as titanium dioxide (TiO₂). TiO₂ is non-toxic and biocompatible, exhibits low optical absorption, is mechanically resistant, and supports high temperature and electrical fields [16], [24]. Several optical fiber sensors based on LPFGs [16], [24]–[29], Mach-Zehnder Interferometers [30], and fiber Bragg gratings [31], mainly fabricated by exposure to UV and CO₂ laser radiation, and electric arc discharge, were coated with TiO₂ thin films given the advantages shown. However, the fs-laser fabrication of LPFGs operating in the TAP with coupling to asymmetric cladding modes and coated with TiO₂ thin films has not been systematically studied.

In this work, the entire fabrication process, including the coating with different TiO₂ film thicknesses, and characterization of the TAP LPFGs in SMF-28e fibers inscribed by fs-laser direct writing, is described. The impact of the fabrication parameters in the coupling behavior was studied in order to improve the coupling efficiency and obtain an LPFG in the TAP condition surrounded by air or water. The change in the effective RI of the asymmetric LP_{1,12} cladding mode as a function of the TiO₂ film thicknesses was evaluated through simulation and experimentally. The resonance wavelength sensitivity to the SRI of the uncoated and TiO₂ coated LPFGs operating at the TAP in air and water was evaluated and compared to the literature's reported works.



Fig. 1. Schematic illustration of the LPFGs: (a) Fabrication, (b) coating, and (c) characterization procedures.

II. MATERIALS AND METHODS

The laser direct writing system assembled employs a fiber amplified fs-laser (Satsuma HP, from Amplitude Systèmes), operating at 515 nm with a pulse duration of \approx 250 fs at a repetition rate of 500 kHz. A 40× aspherical lens (Newport 5722-A-H) with a numerical aperture of NA = 0.5 was used for focusing the laser beam inside the SMF-28e Corning fiber core. The lens was mounted in a Y (Newport M-ILS150CC) and Z (Newport VP-25XA) precision linear stages, which allow motion in the direction transversal to the fiber axis and the beam propagation direction, respectively. A high-quality X air-bearing linear stage (Aerotech ABL10100-LN) with a resolution of 0.5 nm was utilized to scan the optical fiber. The laser beam polarization was adjusted to be parallel to the scanning X direction, which is parallel to the fiber axis. The optical fiber without coating was held by two clamps, where each one is part of a 3-axis positioner, mounted in the X-stage. The optical fiber was stretched and kept under constant tension (1 N) during the fabrication. The real-time monitoring and the adjustment of the fabrication tension were realized through a calibrated load cell (Interface SML series). The precise positioning of the grating in the fiber core was realized using the image on the CCD camera acquired through the writing lens, as illustrated in Fig. 1(a). The focal positions within the fiber were adjusted through the back-reflected laser beam from the fiber surface, tracking a single bright spot when the laser focus is positioned at the top center position of the fiber cladding

For writing TAP LPFGs, the laser gate was externally controlled by a periodic square time function with a duty cycle of 50% generated by the synthesized function generator (DS345, Stanford Research Systems). Writing of gratings was achieved by translating the optical fiber at constant velocity (v) along X-axis, with the signal modulation turned on [11].

The spectral characteristics of the LPFGs were monitored in real-time by using an unpolarized broadband light source (BBS) and an Optical Spectrum Analyzer (OSA) model ANDO AQ-6315B, following the procedure described in [11].

The LPFGs were fabricated with different modulation periods, a 50 μ m/s scan velocity, a fiber tension of 1 N, 40 mm long and with pulse energies of 120 and 140 nJ at a depth of 60 and 57 μ m from the top center position of the fiber cladding, respectively. The LPFGs were annealed, according to the procedure reported in [11] before the TiO₂ thin film coating to ensure that the wavelength and the intensity of the resonance band remain unchanged with the temperature used in the deposition procedure.

The TiO₂ thin-film deposition around the TAP LPFG was done by thermal evaporation of pure titanium in a controlled oxygen atmosphere using an electron beam evaporator (Auto 306, Edwards Ltd, U.K.), following the procedure described in [19], [20], producing homogeneous TiO₂ films with thicknesses of 10, 20, and 30 nm, as illustrated in Fig. 1(b).

A RI characterization setup was prepared to measure the LPFGs spectrum using different surrounding mediums. The LPFGs were characterized in the transmission mode, keeping the fiber stretched at a constant tension produced by a suspended weight of 5.8 g and measuring the BBS normalized signal with the OSA without polarization control from 1200 to 1700 nm with a 2 nm resolution.

The LPFGs were introduced in a glass V-groove, parallel aligned to the fiber axis, and characterized with RI calibrated oils (Cargille-Sacher Laboratories Inc., USA), as illustrated in Fig. 1(c). The RI oils used belongs to the series AAA $n_D^{25^{\circ}C}$ = 1.3000 - 1.3950 ± 0.0002 and AA $n_D^{25^{\circ}C}$ = 1.4000 - 1.4580 ± 0.0002. After each measurement, both LPFG and the V-groove were cleaned with acetone to avoid contamination of the next SRI oil. The measurements were performed in a temperature-controlled environment, monitoring the temperature during the characterization with a type K thermocouple thermometer (AMECaL ST-9612).

To assess the stability and repeatability of the sensing scheme, the spectral resolution was calculated based on the method reported in [32]. The RI spectral resolution, *RRI*, was estimated considering the following expression [32]:

$$R_{RI} = \frac{\rho\sigma}{S_{RI}} \tag{2}$$

where ρ represents the confidence interval (99.7%), σ is the standard deviation of a set of measurements performed in repeatable and reproducible conditions associated with a step variation of the external RI, and S_{RI} is the sensitivity linked with the SRI measurement range.

III. EXPERIMENTAL RESULTS AND DISCUSSION

A. Characterization of the TAP LPFGs Writing

Two 40 mm long LPFGs with a period of 191.8 μ m were fabricated at a depth of 60 and 57 μ m from the top center position



Fig. 2. Optical microscope views of the cross-sections of the fs-laser written LPFGs in the SMF-28e fiber (8.2 μ m core diameter) at a depth from the fiber surface of (a) 60 μ m with pulse energy of 120 nJ and (b) 57 μ m with pulse energy of 140 nJ.

of the fiber cladding with pulse energies of 120 and 140 nJ, respectively.

The shape of the fs-laser induced RI modification was assessed by cleaving the optical fibers and inspecting with an optical microscope (Leica DM6000 M) the cross-sections of the structures, as shown in Fig. 2.

Both types of fs-laser induced RI modifications are transversely inhomogeneous and asymmetric, resembling an elliptic and rectangular shape that extends across the core center, as shown in the insets of Fig. 2(a) and (b), respectively. The non-uniformity and localization of the RI modifications lead to coupling to the asymmetric LP_{i,j} cladding modes of order *j*. For LPFGs written with periods between 175 and 200 μ m coupling occurs to the asymmetric LP_{1,12} cladding mode, accordingly with the simulated phase-matching curves reported in [6], and as addressed in a previous work [11].

LPFGs with periods between 190 and 193 μ m were inscribed with the fabrication parameters aforementioned, to study the spectral behavior at, or very close to, the TAP of the asymmetric $LP_{1,12}$ cladding mode, considering the LPFGs surrounded by air and water. The LPFGs transmission spectra in air and water for the two fabrication conditions are shown in Fig. 3(a) and (b), respectively. For the grating period of 190 μ m, the LP_{1,12} mode exhibits two resonances at ${\sim}1290.8$ and ${\sim}1600.2$ nm $(\Delta \lambda = 309.4 \text{ nm})$ in air, as shown in Fig. 3(a). When the grating inscription period increased from 190 to 191.7 μ m, the two resonances approach, reaching a separation of 51.4 nm. As can be seen in Fig. 3(c), the 191.8 μ m period matches the peak of the dispersion curve of the $LP_{1,12}$ cladding mode, where the two resonance bands in the transmission spectrum merge into one band at \sim 1442.7 nm. Moreover, the increase of the inscription period to 192 μ m results in a decrease of coupling efficiency, but not in wavelength, as shown in Fig. 3(c) and (d). For LPFGs fabricated with periods greater than 192 μ m and surrounded by air, the resonance vanishes because the coupling occurs outside of the dispersion curve of the LP_{1,12} cladding mode. However, if the SRI increase, i.e., when the LPFGs are immersed in water, the coupling behavior changes, which induce a re-distribution of the cladding modes. Thus, for the LPFGs fabricated with 192 μ m and immersed in water, the LP_{1,12} mode exhibits two resonances around the TAP region at \sim 1374.4 and \sim 1523.7 nm



Fig. 3. Transmission spectra of the LPFGs fabricated at a depth of (a) 60 μ m with pulse energies of 120 nJ and (b) 57 μ m with pulse energies of 140 nJ; (c) resonance wavelengths; (d) optical power versus the grating period for fabrication at a depth of 60 μ m with a pulse energy of 120 nJ.

 $(\Delta \lambda = 149.3 \text{ nm})$. The LPFGs operation at the TAP in water was achieved with a grating inscription period of 192.5 μ m. In summary, for the grating periods of 191.8 and 192.5 μ m, it was verified that the LP_{1,12} mode exhibits a TAP at ~1442.7 and ~1448.6 nm in air and water, respectively.

In order to improve the amplitude of the LPFGs attenuation band, the fs-laser pulse energy was increased to 140 nJ, and the laser beam focus adjusted at a depth of 57 μ m from the top center position of the fiber cladding. This procedure allowed the RI modification across the entire core center, as shown in the inset of Fig. 2(b). The difference between the effective RIs of the core and cladding modes increased, and the underlying RI modification induced a change in the coupling behavior leading to a re-distribution of the cladding modes. For this writing condition, the LPFGs operation at the TAP was achieved with grating inscription periods of 192 μ m in air and 193 μ m in water, resulting in resonance wavelengths at \sim 1451.1 and \sim 1453.8 nm, respectively. In addition, the coupling efficiency was improved for the 192 μ m LPFG in air, resulting in an -3.1 dB increase of the attenuation band, in comparison with the attenuation band value ($I_{res} = -13.0$ dB) of the 191.8 μ m LPFG. However, as can be seen in Fig. 3(b), additional peaks appear on the LPFGs resonance for this writing condition. These additional peaks can be due to the coupling to the higher-order cladding modes, which rely on the sharp inhomogeneities as well as the position and asymmetrical shape of the induced RI modification. In [33], it was theoretically and experimentally determined that the additional peaks were mainly caused by decentered and homogeneously RI modification induced in the fiber core. The grating inscription with an offset of 0.8 μ m on the X-axis makes coupling occurs to the asymmetric higher-order cladding modes, where the additional peaks appear between the main resonances. The suppression of these additional peaks can be achieved inducing homogeneous and centered RI modifications in the fiber core, which results in the coupling to the symmetric cladding modes, as reported in [33], [34].



Fig. 4. Fs-laser TAP LPFGs inscribed with a period of 191.8 μ m and numbered from 1 to 12.

 TABLE I

 MAIN CHARACTERISTICS OF THE SET OF LPFGS FABRICATED BY FS-LASER

| Values | λ_{res} (nm) | Intensity (dB) | FWHM (nm) |
|--------------------|----------------------|----------------|-----------|
| Average | 1442.7 | -7.6 | 115.2 |
| Standard Deviation | 1.0 | 0.4 | 3.5 |

As can be seen in Fig. 3(a), the fabrication of TAP LPFGs without additional peaks in the transmission spectra and coupling to asymmetric modes was achieved due to the position, asymmetry, and elliptic shape of the induced RI modification, depicted in the inset of Fig. 2(a). LPFGs fabricated with longer periods, which allow coupling to the asymmetric LP_{1,6} cladding mode, without additional peaks in the transmission spectrum, were also reported in our previous works [11], [35].

In order to assess the reproducibility of the fs-laser direct writing, twelve LPFGs operating at the TAP region were fabricated with the same fabrication conditions. Figure 4 shows the transmission spectra of twelve TAP LPFGs fabricated with a period of 191.8 μ m.

The wavelength, the intensity, and the full width at half maximum (FWHM) of the resonance bands are summarized in Table I. Accordingly, it may be claimed the reproducibility of the fabrication process. The intensity reproducibility was the most challenging parameter to achieve. However, the variation in intensity between devices is not critical for RI sensing. Precise fabrication of fs-laser LPFGs operating at the TAP relies on the sub-micron period control, pulse energy, focal position of the laser beam as well as the fiber tension during fabrication.

The LPFGs sensitivity to the SRI could be further improved by selecting the optimum TiO_2 film thickness for a specific cladding mode. Thus, the behavior of the effective index of the asymmetric LP_{1,12} cladding mode as a function of the TiO_2 film thicknesses was characterized through a 2D analysis of the optical fiber structure and the electromagnetic field modes using the mode analysis of the COMSOL Multiphysics.

The optical fiber structure (SMF-28e, Corning) with four concentric layers was simulated, and the parameters value are presented in Table II. The Sellmeier coefficients for germanium-doped silica [36] and silica [37] were used to calculate the core and cladding RIs at 1442.7 nm, respectively, given by the

TABLE II FIBER PARAMETERS USED IN THE SIMULATION



Fig. 5. Simulated, (a) mode profiles (electric field norm) for the $LP_{0,1}$ and $LP_{1,12}$ modes, respectively, considering the LPFG surrounded by air and (b) effective RI of the $LP_{1,12}$ cladding mode as a function of the TiO₂ film thickness for a SRI of 1.4300.

Sellmeier relation reported in [36]. According to DeVore's empirical formula [38], the refractive index of TiO_2 film thickness at 1442.7 nm was determined.

The fs-laser induced RI variation (resembling an elliptic depicted in the inset of Fig. 2(a)), in relation to the core, of the reduced (located above) and increased (located under) RI regions were set to -0.025 and 0.009, respectively.

These values were determined by the measurements of the RI profile at 980 nm with an IFA-100 (Interfiber Analysis) interferometric system. The dimension used in the structure design tried to represent as close as possible the fs-laser induce RI modification cross-section.

The mode profiles of the LP_{0,1} and LP_{1,12} modes, at a wavelength of 1442.7 nm were evaluated, as shown in Fig. 5(a), yielding effective mode indices of ~1.44599 and ~1.43847, respectively. These values yield a grating period of 191.8 μ m given by equation 1, which was corroborated by the experimental results, where for the same inscription period, the coupling occurs to the asymmetric LP_{1,12} cladding mode.

Fig. 5(b) shows the variation of the effective RI of the $LP_{1,12}$ mode as a function of the TiO₂ film thickness for a SRI of 1.4300. The simulation was performed for a SRI closer to the cladding material to increase the interaction between the cladding mode and the external medium. The transition region occurs approximately in the range between 40 to 60 nm, as shown in Fig. 5(b). Otherwise, for TiO₂ film thicknesses above approximately 60 nm, the effective RI change stabilizes. However, in a more detailed analysis, the highest effective RI change

as a function of the TiO₂ film thickness can be observed for a thickness between 40 to 50 nm. Therefore, for LPFGs fabricated with periods between 198.6 to 199.5 μ m, and operating at the TAP in an SRI of 1.4300, the optimum TiO_2 film thickness is between 40 to 50 nm. The evolution of the effective RI as a function of TiO₂ film thickness observed is similar to the one reported in [24], [39], [40]. The behavior of the symmetric $LP_{0,10}$ cladding mode, as a function of the TiO_2 film thickness was verified by simulation and experimentally in [16], and the transition region is located between 60 to 80 nm. Thus, the transition regions rely on the grating fabrication period, the order of the coupling mode, and the SRI of interest underlying the film thickness [16], [24], [25]. The simulation (COMSOL Multiphysics software, version 5.3a) for TiO_2 film thicknesses lower than 40 nm was hampered by compilation issues concerning the minimum mesh size. Consequently, the experimental results demonstrated here could not be replicated theoretically. The mesh was defined by creating free triangular nodes with an extremely fine element size. However, this mesh does not adapt well to the curved geometry of the TiO₂ film for thicknesses lower than 40 nm, leading to inaccurate results (apparently, recent versions of the software used tackle this issue). Experimentally, the fabricated devices could use thicker TiO₂ films at a cost of a lower sensitivity since the resonances would move away from the TAP. Therefore, the devices can be optimized by increasing the film thickness and adjusting the grating period accordingly in order to maintain the grating very close to the TAP, thus ensuring high sensitivity.

The high RI of TiO₂ film coating promotes a quicker transition between cladding guided modes in overlay guided modes [41]. Therefore, LPFGs fabricated with periods to operate at TAP in air and in water were coated with TiO₂ thin films with a thickness of 10, 20, and 30 nm. The thickness of the coated LPFGs was selected to allow the operation very close to the TAP. This causes a coupling behavior change, which leads to a wavelength and intensity shift of the TAP LPFGs resonances. Table III presents the wavelength and intensity shifts for each LPFGs after coating. The resonance band split into two after coating, and the separation between the two resonances increases with the TiO_2 film thickness. Furthermore, a decrease of the attenuation band was observed when the TiO2 thicknesses increased, more pronounced for the attenuation band $(I_{\lambda 1})$ of the 192.5 μ m LPFG. The wavelength and intensity shifts also depend on the slope of the characteristic curve of the resonance band.

B. Refractive Index Sensing

The wavelength sensitivity to the SRI, between 1.0000 and 1.4480, was assessed for the LPFGs fabricated with periods of 191.8 and 192.5 μ m, uncoated (bare), and also coated with TiO₂ film thickness of 10, 20, and 30 nm. For clearness, few of the acquired spectra are shown in Fig. 6(a) and (b) for the uncoated 191.8 and 192.5 μ m LPFGs, and in Fig. 6(c) and (d) for the 191.8 and 192.5 μ m LPFGs coated with 10 and 20 nm thick TiO₂ films, respectively.

As can be seen in Fig. 6, the separation between the two resonances increases with the increase of the TiO₂ thickness, and with the SRI. The LPFGs fabricated with a period of 192.5 μ m allows the measurement of a broader RI range, which is restricted

TABLE III SUMMARY OF THE SPECTRAL CHARACTERISTICS OF THE TIO2 TAP LPFGS

| SRI | Λ (µm) | 1 (2020) | I_{res} (dB) | TiO ₂ (nm) | $\Delta\lambda_{res}$ (nm) | | ΔI_{res} (dB) | |
|--------|----------------|----------------------|----------------|-----------------------|----------------------------|-------------|-----------------------|-----------------|
| | | λ_{res} (nm) | | | λ_{I} | λ_2 | $I_{\lambda I}$ | $I_{\lambda 2}$ |
| 1.0000 | 101.9 | 1442.7 | -10.6 | 10.0 | -64.3 | 52.0 | -1.3 | 1.9 |
| | 191.0 | | | 20.0 | -77.0 | 63.9 | -0.5 | 3.2 |
| 1.3330 | 192.5 | 1448.6 | -15.6 | 10.0 | -36.3 | 42.4 | 1.8 | -1.9 |
| | | | | 20.0 | -55.6 | 58.3 | 2.8 | -2.1 |
| | | | | 30.0 | -88.8 | 95.2 | 4.7 | -0.4 |



Fig. 6. Transmission spectra of LPFGs fabricated with a period of 191.8 and 192.5 μ m, (a) and (b) uncoated, and (c) and (d) coated with 10 and 20 nm thick TiO₂ film, respectively for several values of the surrounding refractive index.

by the wavelength measurement range, in this case, up to SRI value of 1.4320 for the both uncoated LPFGs. For the coated LPFGs, the dual-wavelength resonance measurement was restricted to SRI values lower than 1.4320. The increase of the coating thickness induces broader wavelength shift for a given SRI, which is more noticeable for the wavelength resonance, λ_2 .

The normalized wavelengths shifts as a function of the SRI, from 1.3300 to 1.4480, are shown in Fig. 7(a) and (b) for the 191.8 and 192.5 μ m LPFGs uncoated and TiO₂ coated, respectively. The wavelength response is not linear for this RI range. Thus, the first derivative of the data plotted in Fig. 7(a) and (b) were calculated to assess the wavelength sensitivity.

In the RI range between 1.3700 to 1.4080, the 191.8 μ m coated LPFGs presents a wavelength sensitivity increase to SRI, larger for thicker coating thicknesses. The dual-wavelength resonance measurement was defined as the total wavelength shift between the two resonances ($\Delta \lambda = \lambda_2 - \lambda_1$).

The LPFG coated with 20 nm TiO₂ thickness, presents a maximum wavelength sensitivity of -1583.5, and 2560.7 nm/RIU for the resonances λ_1 and λ_2 , respectively. Therefore, a sensitivity value of 4144.2 nm/RIU was reached at a RI of 1.4040.

The 191.8 μ m LPFG coated with 10 nm thick TiO₂ film, reached a wavelength sensitivity of 6351.1 nm/RIU ($\lambda_1 =$ -1611.6, and $\lambda_2 =$ 4739.5 nm/RIU) at a RI of 1.4200. However, the maximum wavelength sensitivity of 8169.8 nm/RIU ($\lambda_1 =$ -2020.6, and $\lambda_2 =$ 6149.2 nm/RIU) at a RI of 1.4320 was found



Fig. 7. Normalized wavelength shift (a) and (b), and wavelength sensitivity (c) and (d) of the uncoated and coated with different TiO₂ films thickness for LPFGs fabricated with a period of 191.8 and 192.5 μ m as a function of the surrounding refractive index.

for the uncoated 191.8 μ m LPFG. For the 192.5 μ m LPFGs a maximum wavelength sensitivity value of 11257.5 nm/RIU $(\lambda_1 = 2876.8, \text{ and } \lambda_2 = 8380.7 \text{ nm/RIU})$ at a RI of 1.4320 was found for the uncoated LPFG. Therefore, it can be seen that the uncoated 192.5 μ m LPFG sensitivity was improved by a factor of \sim 1.4 in comparison to the 191.8 μ m LPFG. However, for the 192.5 μ m LPFG, it was seen a wavelength sensitivity decrease with the increase of the TiO₂ coating thickness for RI higher than 1.4120. This was due to the impossibility of dual-wavelength resonance measurement (resonance λ_2 outside of the measurement range). As such, the LPFG coated with 10 nm TiO₂ thickness, reached a wavelength sensitivity of 9117.1 nm/RIU ($\lambda_1 = -2580.8$, and $\lambda_2 = 6536.3$ nm/RIU) at a RI of 1.4280. Regarding the LPFG coated with 20 nm TiO₂ thickness, a wavelength sensitivity of 8347.4 nm/RIU ($\lambda_1 =$ -2553.4, and $\lambda_2 = 5794.0$ nm/RIU) at a RI of 1.4240 was achieved. In the RI between 1.3700 to 1.4120 (upper limit for dual-wavelength measurement) for all LPFGs, the maximum wavelength sensitivity of 8051.4 nm/RIU ($\lambda 1 = -3773.9$, and $\lambda 2 = 4277.5$ nm/RIU) was achieved for the LPFG coated with 30 nm of TiO_2 . It can be seen that the sensitivity enhancement depends on the fabrication period, which allows operating closer to the TAP region at a specific RI, where the sensitivity reaches maximum values. Furthermore, for SRI closer to the cladding RI of the SMF-28e fiber, i.e., $n_D^{25^{\circ}C} = 1.4540$, cladding modes interact more with the external medium, leading to the increase

 $1 PEG \Lambda = 191 8 \mu m$ LPFG Λ = 192.5 μm Wavelength Shift (nm) 200 (b) a (a) 100 ro 0 -100 Norm. -200 1.35 1.37 1.33 1.35 1.36 1.37 1 33 1.34 1.36 1.34 Refractive Index Refractive Index Uncoated: $\lambda_1 \bullet \lambda_2 \Box$ TiO₂ (nm): $\lambda_1 \bullet 10 \bullet 20 = 30$ λ_2 o 10 \triangle 20 \bigtriangledown 30

Fig. 8. Normalized wavelength shift (a) and (b) for the uncoated LPFGs and coated with different thick TiO₂ films fabricated with a period of 191.8 and 192.5 μ m, respectively in the RI range of water-based solutions. Experimental data points were fitted to a linear equation.

TABLE IV WAVELENGTH SENSITIVITY FOR BARE AND TIO_2 COATED LPFGs in the RI RANGE OF WATER-BASED SOLUTIONS

| Λ | TiO ₂ | SENSITIVIT | LINEARITY (R ²) | | | |
|--------------|------------------|--|------------------------------------|-------------|-------------|--|
| (µm) | (1111) - | λ_1 | λ_2 | λ_1 | λ_2 | |
| 191.8 | Bare | -421.4 ± 27.9 $\Delta \lambda = 1234$ | 813.0 ± 57.4 4.4 ± 72.2 | 0.99 | | |
| | 10.0 | $-437.6 \pm 58.6 \qquad 827.3 \pm 84.8$ | | 0.95 | | |
| | | $\Delta\lambda = 1264$ | 0.93 | | | |
| | 20.0 | -487.0 ± 53.7 | 971.0 ± 85.6 | 0.96 | 0.98 | |
| | | $\Delta \lambda = 1458$ | 0.97 | | | |
| | Bare | -1571.9 ± 90.6 | 1579.9 ± 99.6 | | | |
| | | $\Delta\lambda = 3151$ | _ | | | |
| - 192.5 - | 10.0 | -1156.3 ± 56.3 | 1382.9 ± 70.4 | - | | |
| | | $\Delta \lambda = 2539$ | 0.00 | | | |
| | 20.0 | -1012.0 ± 39.5 | 1325.0 ± 54.8 | 0.99 | | |
| | | $\Delta\lambda = 233^{2}$ | | | | |
| | 30.0 | -1001.5 ± 50.8 | 1319.3 ± 65.7 | - | | |
| | | $\Delta\lambda = 2320$ | 0.8 ± 115.4 | | | |
| | | | | | | |

of the LPFG sensitivity. However, the increase of the LPFG sensitivity for the RI range between 1.3700 - 1.4120, was mainly caused by the increase of the TiO₂ film thickness, which enhanced the evanescent wave interaction with the surrounding medium.

The normalized wavelengths shift to SRI in the range between 1.3300 to 1.3700 (i.e., around water-based solutions) was also determined, as depicted in Fig. 8. Although the wavelength response to the SRI presents a nonlinear trend, in a narrow SRI range it can be considered linear. Thus a linear fit was applied to the results in Fig. 8 in order to assess the wavelength sensitivity (average \pm standard deviation), presented in Table IV.

For the 191.8 μ m LPFGs, the wavelength sensitivity in the RI range of water-based solutions was improved, being higher for larger TiO₂ film thickness. As such, a maximum average wavelength sensitivity of 1458.0 ± 137.2 nm/RIU ($\lambda_1 = -487.0 \pm 53.7$, and $\lambda_2 = 971.0 \pm 85.6$ nm/RIU) was achieved for the LPFG coated with 20 nm TiO₂ thickness.

For the 191.8 μ m LPFGs operation occurs far away from TAP for SRI around water solutions. In this zone of the phasematching curve, it was found that the TiO₂ coating improves the wavelength sensitivity. Thus, the proper choice of the LPFGs period allows the tuning of the operation at the TAP, which



Fig. 9. Normalized wavelength shift as a function of time when the surrounding medium undertakes step variations for the resonance: (a) and (b) λ_1 , and (c) and (d) λ_2 of the LPFGs fabricated with a period of 191.8 and 192.5 μ m, respectively.

TABLE V RI RESOLUTIONS FOR THE BARE AND TIO₂ COATED LPFGS

| Λ | TiO ₂ | \$TED - | RI RESOLUTIONS | | |
|---------|------------------|---------|-----------------------|----------------------|--|
| (µm) | (nm) | STEP | λ_1 | λ_2 | |
| 101.0 | Bare | 1 | 1.7×10^{-4} | 2.7×10^{-4} | |
| | | 2 | 2.3×10^{-4} | 5.9×10^{-4} | |
| 191.8 - | 10.0 | 1 | 5.7×10^{-4} | 2.7×10^{-4} | |
| | | 2 | 4.3×10^{-4} | 3.1×10^{-4} | |
| | Bare | 1 | 1.3×10^{-5} | 4.3×10^{-5} | |
| 102.5 | | 2 | 8.2×10^{-5} | 6.8×10^{-5} | |
| 192.5 - | 20.0 | 1 | 8.8×10^{-5} | 8.2×10^{-5} | |
| | | 2 | 6.9×10^{-5} | 5.6×10^{-5} | |

improves the wavelength sensitivity of the bare and coated LPFGs.

However, for the 192.5 μ m LPFGs, it was found that the wavelength sensitivity decreases with the increase of the TiO₂ coating. Indeed, a maximum average wavelength sensitivity of 3151.8 ± 177.0 nm/RIU ($\lambda_1 = -1571.9 \pm 90.6$, and $\lambda_2 = 1579.9 \pm 99.6$ nm/RIU) was achieved for the bare 192.5 μ m LPFG. In addition, considering a narrow RI between 1.3300 to 1.3400, the bare 192.5 μ m LPFG reaches a wavelength sensitivity of 4294.5 nm/RIU ($\lambda_1 = -2038.0$, and $\lambda_2 = 2256.5$ nm/RIU).

The wavelengths sensitivity achieved for each LPFG relies on the TiO_2 coating thickness, which by changing the effective indices of the cladding modes induces a shift in the wavelength position of each TAP LPFG. Therefore, it causes the single resonance band to split into two distinct resonances and, consequently, the sensitivity of the TAP LPFGs decreases.

The choice of a 192.5 μ m fabrication period for an uncoated LPFG allowed the operation at the TAP for RI around waterbased solutions, enhancing the wavelength sensitivity to the SRI. The fine-tuning of the LPFG fabrication period to values higher than 192.5 μ m, in conjunction with an adequate choice of the

TABLE VI COMPARISON BETWEEN THE SENSITIVITY DISPLAYED BY LPFGS OPERATING AROUND TAP REPORTED IN THE LITERATURE AND THIS WORK

| | FABRICATION PROCESS | SENSITIVITY (NM/RIU) | RI RANGE | References | | |
|----------------|--|----------------------|-----------------|------------------|--|--|
| 1. 2. | EXCIMER LASER POINT BY POINT EXPOSURE; CHEMICAL ETCHING. | 6269.2 | 1.3300 - 1.3400 | [42,43] | | |
| 1. | EXCIMER LASER AND AMPLITUDE CHROMIUM MASK; | 6200.0 | 1.3400 | | | |
| 2. | CHEMICAL ETCHING; | 4300.0 | 1.3600-1.4100 | - [24] | | |
| <u> </u> | EXCIMENTASER AND AMPLITUDE CHROMIUM MASK | 22000.0 | 1 2244 1 2255 | | | |
| 2. | CHEMICAL ETCHING: | 22000.0 | 1.3344 - 1.3355 | - [48] | | |
| 3. | COATING WITH DIAMOND-LIKE CARBON. | 2000.0 | 1.3400 - 1.3560 | | | |
| THEO | DRETICAL WORK: | 3750.0 | 1.3300 - 1.3500 | | | |
| 1. 2. | CHEMICAL ETCHING; COATING WITH THIN-FILM. | 143000.0 | 1.3300-1.3310 | [44] | | |
| 1. 2. 3. | Excimer laser point by point exposure; Chemical etching; Coating with poly allylamine hydrochloride/poly acrylic acid. | 5602.0 | 1.3700 - 1.4100 | [45,47] | | |
| 1. 2. | EXCIMER LASER POINT BY POINT EXPOSURE; SOL-GEL BASED TITANIA-SILICA THIN FILM OVERLAY. | 7075.3 | 1.3330-1.3340 | [25] | | |
| 1. 2. 3. | Chemical etching; Coating with Tin Dioxide (SNO ₂). Periodic Laser ablation. | 6430.0 | 1.3740 - 1.3890 | [7] | | |
| Elec | TRIC ARC DISCHARGES | 590.0 | 1.3300 - 1.4200 | [6] | | |
| Fem | FOSECOND LASER DIRECT WRITTEN | 930.0 | 1.3330 - 1.3980 | [46] | | |
| 1. FI 2. Ce | | 3151.7 | 1.3300 - 1.3700 | – This work – | | |
| | Femtosecond Laser direct written: | 4294.5 | 1.3300 - 1.3400 | | | |
| | COATING WITH TIO ₂ . | 11257.4 | 1.4320 | | | |
| | | 8051.4 | 1.3700 - 1.4120 | | | |

This comparison was restricted to the LPFGs operating around the TAP and fabricated in the SMF-28 fiber.

 TiO_2 film thickness, will allow the operation at TAP for SRI around water-based solutions.

The analysis of the spectral resolution was done as described in the section material and methods, and the data concerning the SRI step variations are shown in Fig. 9. Table V presents the calculated RI resolutions for the bare and TiO₂ coated LPFGs. Refractive index resolutions with magnitudes of 10^{-4} and 10^{-5} RIU were achieved for the LPFGs fabricated with a period of 191.8 and 192.5 μ m, respectively.

Finally, a comparison between the LPFGs fabricated in SMF-28 fibers operating at the TAP reported in the literature and the ones developed in this work was realized and summarized in Table VI.

Thus, among the few LPFGs fabricated in SMF-28 fibers reported operating at the TAP, the LPFGs fabricated on this work exhibited higher sensitivity in comparison to the LPFGs fabricated by electric-arc, fs-laser direct written and also excimer laser exposure.

Nevertheless, the maximum sensitivity (i.e., 143000 nm/RIU for a very narrow RI range between 1.3300 to 1.3310, [44]) was theoretically achieved for the TAP LPFGs with reduced cladding diameter and coated with a thin film. Experimentally, a sensitivity over 22000 nm/RIU for a very narrow RI range between 1.3344 to 1.3355 was reported for the TAP LPFGs with reduced cladding diameter and coated with a diamond-like carbon thin

film [48]. The demonstrated LPFGs sensitivity enhancement relies mainly on the evanescent interaction achieved by reducing the cladding diameter. However, this approach involves a complex fabrication process to tune the LPFG operation at the TAP and makes the optical fiber fragile and difficult to handling. The fabrication process proposed in this work allows optimizing the LPFGs sensitivity to the SRI without making the optical fiber fragile in comparison to the fabrication process using chemical etching.

Indeed, the increase of the LPFGs sensitivity for SRI closer to 1.4300, mainly caused by the increase of the TiO_2 film thickness, makes them suitable, for example, for the detection of fuel adulteration (kerosene in petrol), which leads to air pollution and the consequent effects on public health. The refractive index of pure petrol and kerosene are 1.4270 and 1.4420, respectively [31]. The LPFGs can also be coated with a polymer engineered to be sensitive to external parameters, and having a RI close to the value where the highest sensitivity was obtained, and that changes with the ambient humidity or the presence of a specific gas, for example [25]. LPFGs operating at the TAP inscribed by a CO₂ laser in B-Ge doped fiber for fuel adulteration detection was reported in [49]. Here, an average grating sensitivity of 1635.0 nm/RIU for SRI in the range between 1.3970 to 1.4372 has been demonstrated. Nevertheless, a maximum wavelength sensitivity value of 11257.5 nm/RIU at a RI of 1.4320 was

reported in our work. Therefore, it can be seen that the LPFG sensitivity was improved by a factor of \sim 6.9 in comparison to TAP inscribed by a CO2 laser [49].

IV. CONCLUSION

The precise production of LPFGs operating at the TAP in an SMF-28e fiber through fs-laser direct writing was demonstrated. The inscription of LPFGs with a clear spectrum without higher-order interference peaks was achieved by writing RI modification with a laser pulse energy of 120 nJ at a depth of 60 μ m from the top center position of the fiber cladding. The LPFGs operating at the TAP of the asymmetric $LP_{1,12}$ cladding mode in air and water were fabricated with an RI modulation period of 191.8 and 192.5 μ m, yielding a resonance wavelength at approximately 1442.7 and 1448.6 nm, respectively. It was demonstrated that the precise fabrication of the fs-laser LPFGs operating at the TAP relies on the sub-micron period control, the pulse energy, the focal position of the laser beam as well as the fiber tension during fabrication. The LPFGs were coated with different TiO₂ film thickness to improve the sensitivity to SRI, and their spectral behavior around the TAP in air and water characterized. The wavelength sensitivity to the SRI was also assessed. Therefore, a maximum sensitivity of ~8051.4 nm/RIU in the range between 1.3700 to 1.4120 was obtained for the 192.5 μ m LPFG coated with 30 nm TiO₂ thickness. The maximum sensitivity in the RI range of water-based solutions was approximately 3151.7 nm/RIU, achieved for the uncoated 192.5 μ m LPFG. The SRI sensitivity could be further improved, tuning the LPFG fabrication period to operate at the TAP accordingly to the TiO_2 film thickness.

Another approach could be to tune the operation at TAP by reducing the cladding diameter and posteriorly coating with different TiO_2 film thickness. Further works are ongoing in order to optimize the fs-laser TAP LPFGs, which will be essential to the suitable application in the biological, chemical, or biochemical sensing applications.

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