

Femtosecond written Long Period Fibre Gratings coated with Titanium Dioxide for improved sensitivity

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ABSTRACT

Long Period Fibre Gratings (LPFGs) were fabricated by femtosecond (fs) laser direct writing in a standard single mode fiber (SMF-28e). Then, LPFGs were optimized with annealing treatment, for the deposition of a titanium dioxide (TiO₂) coating by physical vapor deposition (PVD) process. Different thin film thicknesses were deposited and the wavelength and optical power of the LPFG attenuation band were measured. The characterization of the spectral response of the femtosecond LPFGs bare, with 30 nm and 60 nm of titanium dioxide thick film to variations of the surrounding refractive index (SRI) was performed achieving sensitivities of ~570 nm/RIU at 1.3600 RIU in the case of the LPFG coated with 60 nm of TiO₂, a 10-fold increase over the corresponding for a bare LPFG. For SRI values higher than the cladding refractive index, a sensitivity over ~3000 nm/RIU was determined for 30 nm of titanium dioxide thick film, a region where the bare LPFGs are useless. A limited range of SRI values can be accessed by using the optical power shift with a bare LPFG. For 30 nm of titanium dioxide, the LPFG is useful from 1.3100 to 1.4320, where the optical power shift is a quasi-linear function of the SRI, with a range of ~10 dB. Moreover, values as high as 50 and 120 dB/RIU at 1.3200 and 1.4200, respectively, can be obtained by choosing the proper film thickness. Preliminary studies revealed that coating femtosecond laser direct writing LPFGs with titanium dioxide improves their performance.

Keywords: Femtosecond laser direct writing, long period fibre gratings, thin film coating, fibre sensors

1. INTRODUCTION

The operation principle of LPFGs relies on the coupling of radiation between the core and co-propagating cladding modes¹ and, therefore, they are intrinsically sensitive to external phenomena, such as mechanical strain and temperature. The grating period, the refractive index (RI) modulation, and the surrounding refractive index (SRI) disturbs the coupling conditions leading to variations in the output spectra², thus making LPFGs sensitive to different parameters [2–5].

Several methods have been developed for LPFGs fabrication in a variety of fibers⁴, such as exposure to UV or CO₂ laser radiation⁶, induced electric-arc discharge⁷, ion beam irradiation², chemical etching⁸, and femtosecond laser written⁹. Femtosecond laser-based fabrication does not require any special fiber pre-treatment³. The induced refractive index change has the possibility of being thermally stable over a broader temperature range after a proper annealing process, compared to UV induced gratings². The fs-laser direct writing technique used in this work opens the possibility to inscribe LPFGs inside the core volume with high spatial resolution due to the underlying non-linear light-matter interaction⁹. The phase-matching condition, which results in coupling of wavelengths between the fundamental core mode and forward propagating cladding modes is given by²:

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

where n_{core}^{eff} is the effective refractive index of the propagating core mode at wavelength λ , and $n_{cl,m}^{eff}$ is the refractive index of the m^{th} cladding mode, respectively. The coupling resonant wavelength λ_{res} , can easily be controlled by simply tuning the grating period, Λ , with its period being defined by the laser beam's modulation frequency, f_{mod} and fiber translation velocity, v along its axis⁹.

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The LPFGs inscribed through the fs-laser exposure have a far greater polarization response and the ability to couple to further sets of cladding modes [3,10,11]. The technique is suitable for automated fabrication of a large number of devices as well to the production of hybrid devices, such as fibre Bragg gratings in tandem with LPFGs¹². The highest sensitivity to the SRI index occurs for values slightly lower than the cladding refractive index (CRI), and it increases for higher-order modes [2,5]. When the SRI is higher than the CRI, there is a considerable sensitivity reduction. It was determined by simulation and experimentally demonstrated that LPFGs coated with a high refractive index thin film induces changes on the typical LPFGs spectral characteristics [13–15].

In this work, a femtosecond laser direct writing system was developed to fabricate LPFGs in SMF-28e fibres. Then, the LPFGs annealing treatment was realized to stabilize their spectra for the deposition of TiO₂ coatings by physical vapour deposition (PVD) process. The whole process used in the fabrication, deposition, and characterization of the LPFGs is described. The LPFGs were coated with a TiO₂ thin film, and the spectral behaviour to variations of the SRI greater and lesser than the CRI was measured and analysed. The experimental results show that coating femtosecond laser direct writing LPFGs with TiO₂ improve their performance.

2. MATERIALS AND METHODS

The fabrication of LPFGs inside a standard single mode optical fiber (SMF-28e, Corning) through localized RI modifications was performed with a femtosecond laser direct writing system. The writing system uses a femtosecond fiber amplified laser (Satsuma HP, from Amplitude Systèmes), emitting a 2nd harmonic beam at 515 nm with a pulse duration of ~250 fs at 500 kHz. The linearly polarized beam was adjusted to be parallel concerning the scanning direction and focused inside the fiber core with a 0.55 NA aspherical lens (New-Focus 5722-A-H). The writing lens is mounted in the Y (Newport (M-)JLS150CC) and Z (Newport VP-25XA) precision linear stages, which allows motion in the direction transversal to the fiber axis and the beam propagation direction, respectively. Before writing, the optical fiber coating was removed around the writing section and mounted on an X air-bearing linear stages (ABL10100-LN), customized with a 3-axis optical fiber positioner holder, providing a scanning direction parallel to the fiber axis. The precise positioning of the grating on the fiber core is realized through the writing lens image, acquired with the CCD camera. The real-time monitoring and the adjustment of the fabrication tension (1 N) are possible due to a load cell (Interface SML series) previously calibrated. The control of the writing parameters of the LPFGs structures was performed by homemade LabVIEW software, which was optimized for the LPFGs fabrication, allowing design flexibility⁹. For writing the LPFGs, the laser gate was externally controlled by a periodic square time function with a duty cycle of 50 % generated by a synthesized function generator (DS345, Stanford Research Systems). This simple arrangement allows the automatic writing of LPFGs by simply translating the optical fiber at a constant velocity, along X, with the signal modulation turned on. The LPFGs were fabricated with a 140 nJ pulse energy, 50 μm/s scan velocity, 2.5 cm of length, a refractive index modulation period of 372.5 μm, fiber tension of 1 N, and the writing beam polarization aligned with the scanning direction. The spectral characteristics of the LPFGs during the fabrication process was monitored with a customized setup. Thus, the broad spectrum of a halogen lamp signal transmitted in the grating was measured with the Optical Spectrum Analyzer (OSA, ANDO AQ-6315B) without polarization control from 1000 nm to 1700 nm with a 10 nm resolution. All spectra are normalized to the transmitted spectrum of the light source. A temperature characterization setup was therefore mounted to annealing the LPFGs after the fabrication. The LPFGs were placed in a tubular oven (Termolab, Portugal), and annealed at constant temperature (300 °C), for a dwell time of 25 minutes⁹.

Titanium dioxide films 30, and 60 nm thick were produced around the grating region by thermal evaporation of pure titanium in a controlled oxygen atmosphere using an electron beam evaporator Auto 306 (Edwards Ltd, U.K.). The equipment is fitted with a homemade rotary system positioned in the vacuum chamber in order to rotate the LPFGs. The rotation velocity was set to 5 rpm, a value which was found to produce homogeneous thin films around the cylindrical fibres, for the range of TiO₂ thicknesses used in this work. A quartz micro balance model FTM5 (Edwards Ltd., U.K.) gives the value of film thickness deposited on a flat substrate in order to calibrate the deposition process¹⁶. The LPFGs before and after the deposition of the thin film were characterized in SRI measurements. The SRI characterization was realized with a set of RI calibrated oils (Cargille-Sacher Laboratories Inc., N.J., U.S.A.), varied from 1.3000 to 1.6400. The grating region was introduced at the glass V-groove, parallel aligned to the fiber axis, and previously prepared with the oil sample to be tested. After each measurement, both optical fiber and the V-groove were cleaned with acetone to avoid contamination to the next oil sample. In the characterization, the optical fiber was stretched with an axial tension produced by a weight of 5.8 g. Besides, the broadband light source (BBS, model ASE2000) signal transmitted in the grating was measured with the OSA (model Yokogawa AQ6370D) without polarization control from 1200 nm to 1700 nm with a

2 nm resolution. All spectra are normalized to the transmitted spectrum of the light source. The described processes are illustrated in Figure 1.

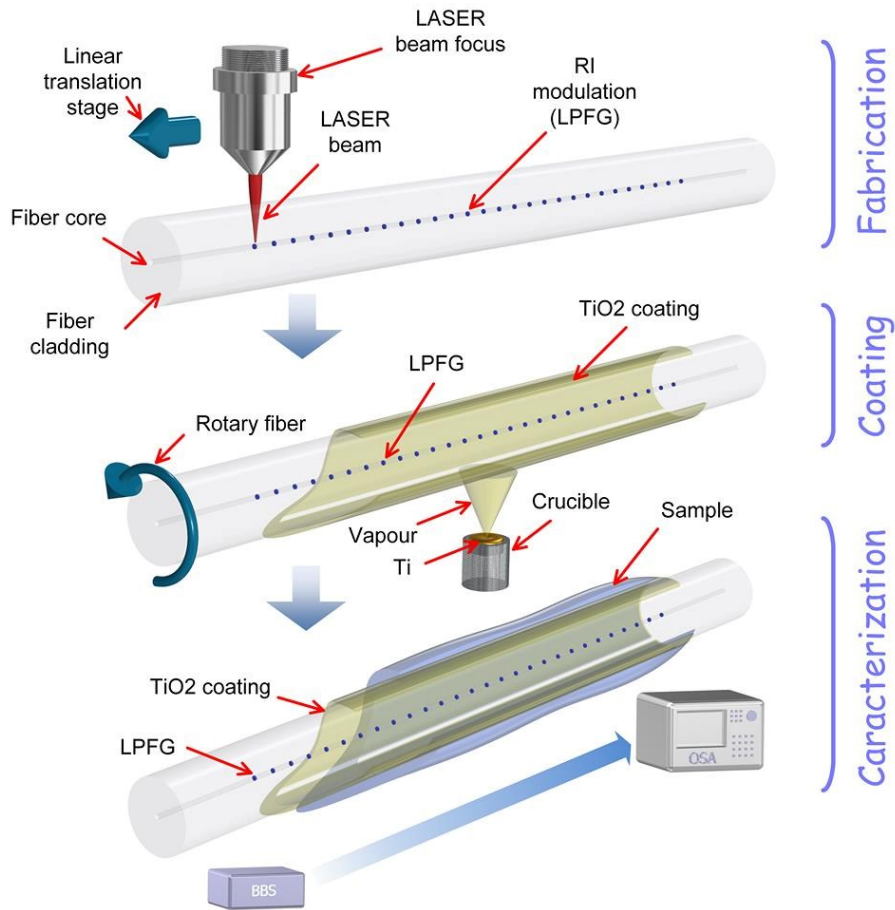


Figure 1. Schematic illustration of the fabrication, coating, and characterization processes of LPFGs.

3. EXPERIMENTAL RESULTS AND DISCUSSION

The transmission spectrums corresponding to the $LP_{1,6}$ cladding mode of three LPFGs, from 1425 to 1575 nm, uncoated (bare) and coated with a 30 and 60 nm thick TiO_2 films with air ($RI = 1.000$) as the SRI are shown in Figure 2.

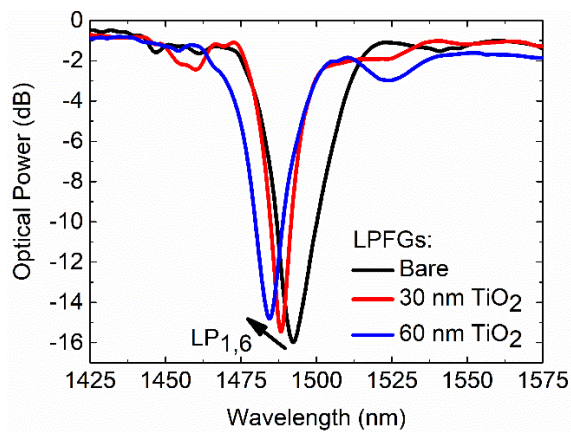


Figure 2. Transmission spectra of the LPFGs, bare and coated with 30 and 60 nm thick TiO_2 films, in the air ($RI = 1.000$).

The coating of the LPFGs with 30 and 60 nm thick TiO_2 films, leads to a shift to shorter wavelengths of the attenuation band of about 4.06 and 7.93 nm, respectively and a decrease of the resonance amplitude, as shown in Figure 3. This behaviour is consistent with the results presented in the literature, for the LPFGs fabricated by the electric arc technique SMF28 fiber, and coated with TiO_2 ¹⁶. The wavelength and amplitude of the attenuation band depend on the effective refractive index of the cladding modes, which in turn depend on the SRI. The thin film induces a re-distribution of the cladding modes that leads to wavelength shift and amplitude intensity variation of the attenuation bands. The transmission spectra of a bare, 30 and 60 nm thick TiO_2 coated LPFGs were measured for SRI in the range 1.0000 to 1.6400, and the recorded spectrums presented in Figure 3 (a) and (b) for the $\text{LP}_{1,6}$ and $\text{LP}_{1,7}$ cladding modes. For clearness, the spectra corresponding to a few SRI values are not shown.

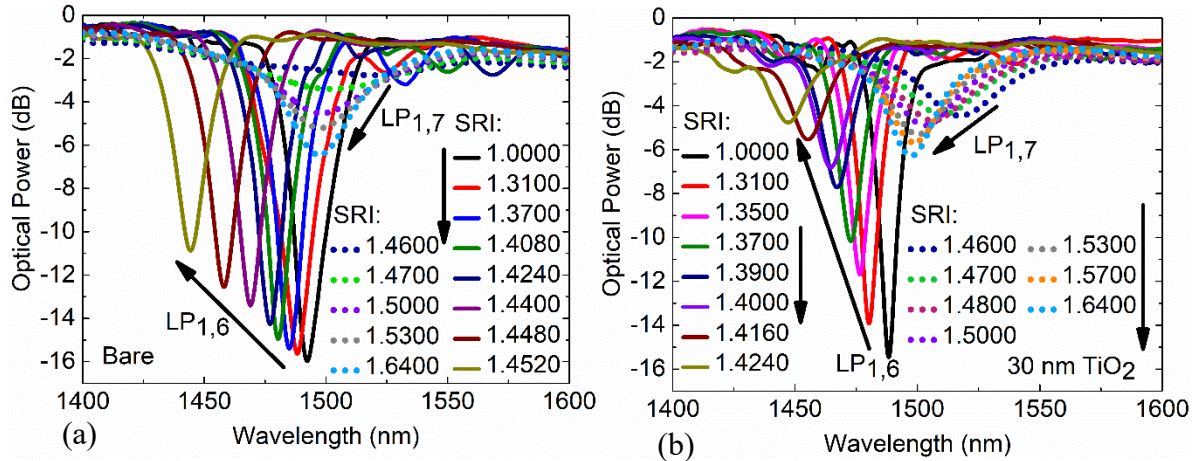


Figure 3. LPFGs transmission spectra ($\text{LP}_{1,6}$ and $\text{LP}_{1,7}$ cladding modes) for several values of the SRI, for: (a) uncoated, and (b) coated with 30 nm thick TiO_2 film.

As the SRI increase from 1.0000 to 1.4520 RIU, the resonance wavelength is gradually blue-shifted, and the intensity loss decrease. However, for SRI higher than the CRI (1.4570), the coated LPFG presents a resonance wavelength shift and intensity loss increase, while the bare LPFG present an intensity loss increase, and is wavelength-independent. The normalized wavelength shift, and the optical power variation to the initial values ($\text{RI} = 1.000$) as a function of the SRI, from 1.3000 to 1.6400, are shown in Figure 4 (a) and (b), for the LPFGs uncoated and coated with 30 and 60 nm thick TiO_2 films, respectively.

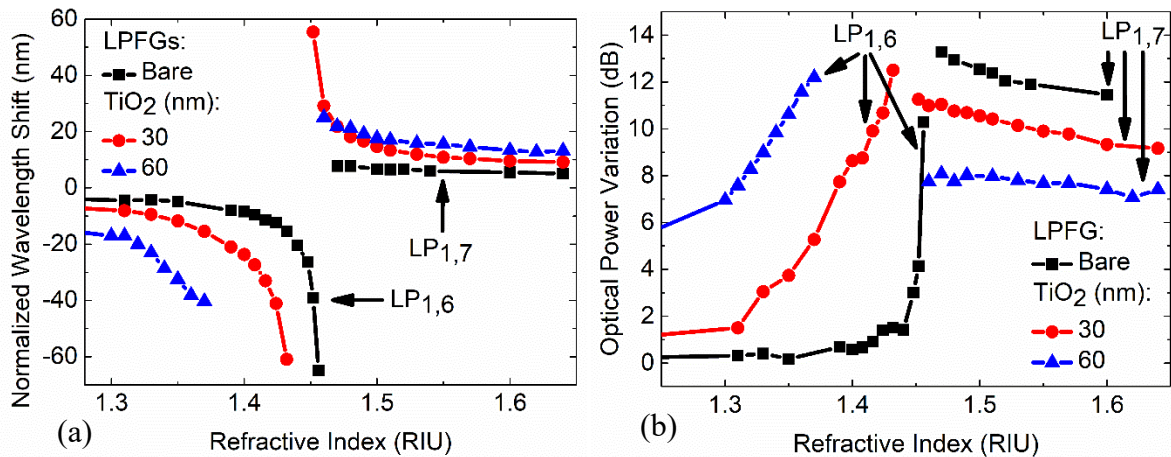


Figure 4. LPFGs uncoated, and coated with 30 and 60 nm thick TiO_2 films: (a) normalized wavelength shift (b), and optical power variation as a function of SRI.

It must be noticed that for SRI values from 1.300 to 1.400, characteristic of many biological materials ¹⁶, the normalized wavelength shift is higher for the TiO_2 coated LPFGs. This is, the higher the film thickness, the higher is the wavelength shift. In this range, a bare LPFG provides very low sensitivity. In addition, for SRI greater than the CRI, the attenuation

bands shift to higher wavelengths, and an increase in the attenuation bands is observed. This behaviour is similar for bare and coated LPFGs. However, the wavelength shift with the SRI is much more noticeable for the coated LPFG due to the higher effective index of the modified cladding layer. The bare LPFG is wavelength insensitive to SRI variations above the CRI, as demonstrated,¹⁶.

The first derivative of the data plotted in Figure 4 (a) and (b) were calculated, to assess the wavelength and intensity sensitivities. The results are shown in Figure 5 (a), (b) and (c).

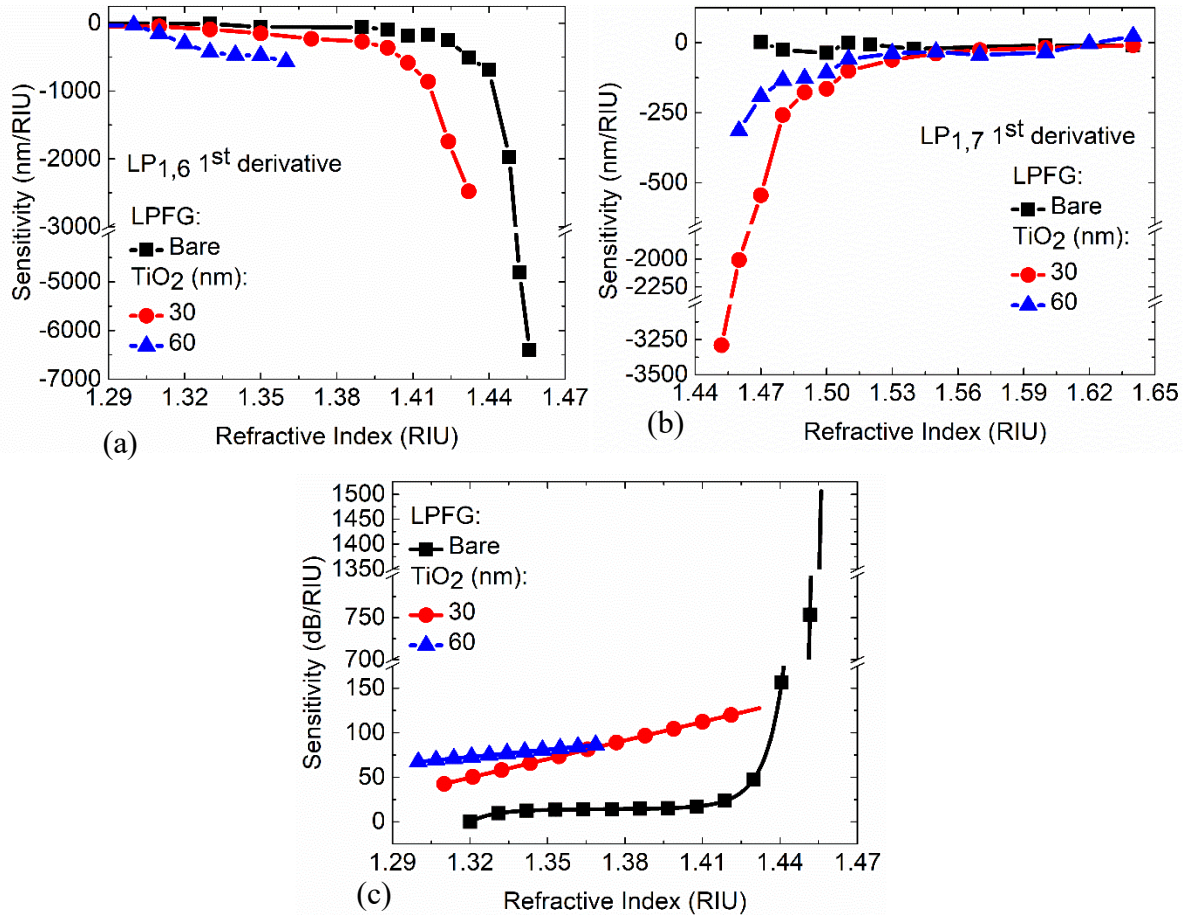


Figure 5. Wavelength sensitivity for (a) LP_{1,6} and (b) LP_{1,7} cladding modes and (c) optical power sensitivity of the LPFGs uncoated, and coated with 30 and 60 nm thick TiO₂ films.

It was observed that the sensitivity increases for higher TiO₂ coating thickness being possible to choose the film thickness according to the SRI that needs to be measured. For example, from 1.3400 to 1.3700 RI range, the average normalized wavelength shift is lower than 60 nm/RIU for the bare LPFG, and ~570 nm/RIU for the LPFG coated with 60 nm TiO₂, as shown in Figure 5 (a). For SRI values higher than the cladding refractive index, a sensitivity over ~3000 nm/RIU was determined for 30 nm of TiO₂ thick film, as shown in Figure 5 (b). From Figure 5 (c) it can be observed a region where a linear fit was obtained for SRI smaller than the CRI, which enables the use of the intensity power variation to measure the SRI variation. However, a limited range of SRI values can be accessed with a bare LPFG. Small optical power changes were verified up to 1.4400. Between this value and 1.4560, a variation of ~9 dB was measured. For 30 nm of TiO₂, the LPFG is useful from 1.310 to 1.432, where the optical power variation is a quasi-linear function of the SRI, with a range of ~10 dB. Moreover, from Fig. 5 (c) values as high as 50 and 120 dB/RIU at 1.320 and 1.420, respectively, can be obtained by choosing the proper film thickness. A limited range of SRI values can be accessed by using the optical power variation with a bare LPFG.

4. CONCLUSIONS

LPFGs were fabricated in an SMF-28e through fs-laser direct writing and coated with TiO₂ thin films of different thicknesses. The coating of the fs-laser direct writing LPFGs with titanium dioxide improved their performance. It was observed an increase of the wavelength and optical power sensitivities over broader ranges of SRI, including for values higher than the CRI. A limited range of SRI values can be accessed by using the optical power variation with a bare LPFG. Compared with the LPFGs fabricated by electric arc discharge, and coated with 60 nm of TiO₂, where a sensitivity of 525 nm/RIU at 1.3600 was reported [16, 17], the fs-laser fabricated LPFGs, and also coated with 60 nm of TiO₂ presented in this work has higher sensitivity, reaching values of ~570 nm/RIU at 1.3600. For SRI values higher than the cladding refractive index, a sensitivity over ~3000 nm/RIU was determined for 30 nm of TiO₂ thick film, a region where the bare LPFGs are useless. For 30 nm of TiO₂, the LPFG is useful from 1.3100 to 1.4320, where the optical power shift is a quasi-linear function of the SRI, with a range of ~10 dB. Given the preliminary results presented, further research is necessary to improve the performance of such fibre based sensors, for instance, optimization of the TiO₂ thickness for a specific SRI range is still to be done, as well as a more thorough optical characterization.

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REFERENCES

- [1] Pal, B. P., [Guided wave optical components and devices: basics, technology, and applications], Academic press (2005).
- [2] James, S. W. and Tatam, R. P., “Optical fibre long-period grating sensors: Characteristics and application,” *Meas. Sci. Technol.* **14**(5) (2003).
- [3] Li, B., Jiang, L., Wang, S., Tsai, H.-L. and Xiao, H., “Femtosecond laser fabrication of long period fiber gratings and applications in refractive index sensing,” *Opt. Laser Technol.* **43**(8), 1420–1423 (2011).
- [4] Ahmed, F., Ahsan, M. S., Lee, M. S. and Jun, M. B. G., “Femtosecond laser based in-fiber long period grating fabrication for improved solution sensing,” *Laser Appl. Microelectron. Optoelectron. Manuf.* XVIII **8607**, 86071L, International Society for Optics and Photonics (2013).
- [5] Coelho, L., Viegas, D., Santos, J. L. and De Almeida, J. M. M. M., “Optical sensor based on hybrid FBG/titanium dioxide coated LPFG for monitoring organic solvents in edible oils,” *Talanta* **148**, 170–176 (2016).
- [6] Nespereira, M., Coelho, J. M. P., Abreu, M. and Rebordão, J. M., “Ultrashort Long-Period Fiber Grating Sensors Inscribed on a Single Mode Fiber Using CO₂ Laser Radiation,” *J. Sensors* **2017** (2017).
- [7] Colaço, C., Caldas, P., Del Villar, I., Chibante, R. and Rego, G., “Arc-induced long-period fiber gratings in the dispersion turning points,” *J. Light. Technol.* **34**(19), 4584–4590 (2016).
- [8] Ascorbe, J., Corres, J. M., del Villar, I. and Matias, I. R., “Fabrication of long period gratings by periodically removing the coating of cladding-etched single mode optical fiber towards optical fiber sensor development,” *Sensors (Switzerland)* **18**(6) (2018).
- [9] Viveiros, D., de Almeida, J. M. M. M., Coelho, L., Vasconcelos, H., Amorim, V. A., Maia, J. M., Jorge, P. A. S., Almeida, J. M. M. M. De, Coelho, L. and Vasconcelos, H., “Spectral Tuning of Long Period Fiber Gratings Fabricated by Femtosecond Laser Micromachining through Thermal Annealing,” *Multidiscip. Digit. Publ. Inst. Proc.* **15**(1), 4 (2019).
- [10] Wolf, A. A., Dostovalov, A. V., Lobach, I. A. and Babin, S. A., “Femtosecond Laser Inscription of Long-Period Fiber Gratings in a Polarization-Maintaining Fiber,” *J. Light. Technol.* **33**(24), 5178–5183 (2015).
- [11] Kondo, Y., Nouchi, K., Mitsuyu, T., Watanabe, M., Kazansky, P. G. and Hirao, K., “Fabrication of long-period fiber gratings by focused irradiation of infrared femtosecond laser pulses,” *Opt. Lett.* **24**(10), 646 (1999).

- [12] Mihailov, S. J., [Femtosecond Laser-Inscribed Fiber Bragg Gratings for Sensing Applications], Elsevier Inc. (2018).
- [13] Del Villar, I., Matias, I. R., Arregui, F. J. and Achaerandio, M., "Nanodeposition of materials with complex refractive index in long-period fiber gratings," *J. Light. Technol.* **23**(12), 4192–4199 (2005).
- [14] Del Villar, I., Zamarreño, C. R., Hernaez, M., Arregui, F. J. and Matias, I. R., "Resonances in coated long period fiber gratings and cladding removed multimode optical fibers: a comparative study," *Opt. Express* **18**(19), 20183 (2010).
- [15] Del Villar, I., Matias, I. R., Arregui, F. J., Lalanne, P., Villar, I. Del, Matias, I. R., Arregui, F. J. and Lalanne, P., "Optimization of sensitivity in Long Period Fiber Gratings with overlay deposition," *Opt. Express* **13**(1), 56–69 (2005).
- [16] Coelho, L., Viegas, D., Santos, J. L., De Almeida, J. and Almeida, J. M. M. M. De., "Enhanced refractive index sensing characteristics of optical fibre long period grating coated with titanium dioxide thin films," *Sensors Actuators B Chem.* **202**, 929–934 (2014).
- [17] Coelho, L., Santos, J. L., Viegas, D. and de Almeida, J. M. M. M., "Fabrication and characterization of metal oxide-coated long-period fiber gratings," *J. Light. Technol.* **34**(10), 2533–2539 (2016).
- [1] Booth, N. and Smith, A. S., [Infrared Detectors], Goodwin House Publishers, New York & Boston, 241-248 (1997).
- [2] Davis, A. R., Bush, C., Harvey, J. C. and Foley, M. F., "Fresnel lenses in rear projection displays," *SID Int. Symp. Digest Tech. Papers* 32(1), 934-937 (2001).
- [3] Van Derlofske, J. F., "Computer modeling of LED light pipe systems for uniform display illumination," *Proc. SPIE* 4445, 119-129 (2001).
- [4] Myhrvold, N., "Confessions of a cybershaman," *Slate*, 12 June 1997, <<http://www.slate.com/CriticalMass/97-06-12/CriticalMass.asp>> (19 October 1997). www.optics4yurresearch.com/7752.html
- [5] Jones, C. J., Director, Miscellaneous Optics Corporation, interview, Sept. 23 2011
- [6] FamilyName, GivenName Initial., "Title," Source, pg# (year).