Femtosecond Laser Direct Writing of Turn Around Point Long Period Fiber Gratings Coated with Titanium Dioxide for Improved Sensitivity

Duarte Viveiros^{1,2,*}, José M. M. M. de Almeida^{1,3}, L. Coelho¹, Helena Vasconcelos^{1,3}, João M. Maia^{1,2}, Vítor A. Amorim^{1,2}, Pedro A. S. Jorge^{1,2} and P. V. S. Marques^{1,2}

¹Center for Applied Photonics, INESC TEC, Rua Dr. Roberto Frias, 4200-465 Porto, Portugal;

²Department of Physics and Astronomy, Science Faculty, University of Porto, Rua do Campo Alegre, 4169-007 Porto, Portugal;

³Department of Physics, School of Science and Technology, University of Trás-os-Montes and Alto Douro, Quinta de Prados, 5001-801 Vila Real, Portugal.

*carlos.d.viveiros@inesctec.pt

Abstract: The fabrication of Turn Around Point Long Period Fiber Grating in standard SMF-28e fibers through femtosecond laser direct writing is demonstrated and its sensing sensitivity is improved with a coating of titanium dioxide through physical vapor deposition. **OCIS codes:** (060.2370) Fiber optics sensors; (060.2430) Fibers, single-mode; (050.2770) Gratings; (290.3030) Index measurements; (280.6780) Temperature; (310.031) Thin films; (320.7090) Ultrafast lasers.

1. Introduction

Long Period fiber gratings (LPFGs) are applied to couple light between the core and co-propagating cladding modes in different types of fibers, such as photonic crystal fibers, standard single-mode optical fibers and photosensitive fibers [1, 2]. This property turns the LPFGs intrinsically sensitive to external perturbations, as mechanical stress, temperature, or refractive index (RI) changes in the surrounding medium [2]. The fabrication of an LPFG with very high sensitivity at a particular wavelength is possible by selecting a cladding mode and period at, or very close to, a turn around point (TAP), also known as phase matching turning point [3, 4]. Each cladding mode exhibits a TAP, at a given period, which corresponds to two resonances wavelengths [3]. Concerning the fabrication and tuning of an LPFG at TAP, several methods have been developed, including exposure to UV and CO_2 laser radiation [5], Gamma radiation [6], taper tuning [6], etching of cladding [6], and overlay coating [1]. However, complex fabrication processes can weaken the optical fiber and render difficult on its implementation in practical applications [2].

The femtosecond (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 interaction [7]. At the phase-matching condition coupling of light between the fundamental core mode and forward propagating cladding modes occurs and is given by $\lambda_{res} = (n_{core}^{eff}(\lambda) - n_{cl,m}^{eff}(\lambda))\Lambda$, where n_{core}^{eff} is the effective RI of the propagating core mode at wavelength λ , $n_{cl,m}^{eff}(\lambda)$ is the effective RI of the m^{th} cladding mode, and Λ is the period of the LPFG [7]. The resonant wavelength can easily be controlled by tuning the grating period which in turn is defined by the laser beam's modulation frequency, f_{mod} and fiber translation velocity v along its axis [7].

In this work, the fabrication of TAP LPFG in an SMF-28e fiber through fs-laser direct writing is demonstrated. The LPFG operating near its phase matching turning point, associated with the $LP_{1,12}$ asymmetric cladding mode, was coated with 20 nm of titanium dioxide (TiO₂) thin-film by physical vapor deposition. The surrounding refractive index (SRI) and temperature measurements were performed for the LPFG before and after the deposition of the thin film to test the sensitivity improvement. The relatively low complexity and the robust nature of the fabricated device open the possibility to chemical and bio-chemical sensing in practical applications.

2. Fabrication and Characterization Setups

The LPFGs fabrication system is mainly composed by a fiber amplified fs-laser (Satsuma HP, from Amplitude Systèmes), a 40× aspherical lens (Newport 5722-A-H) with a numerical aperture of 0.5, three precision linear stages, a synthesized function generator (DS345, Stanford Research Systems) and a CCD camera. The second harmonic beam at 515 nm, with a pulse duration of ~250 fs at a repetition rate of 500 kHz of the fs-laser, was selected for the LPFGs fabrication [7]. The writing lens is mounted in the Y (Newport (M-)ILS150CC) 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. The X air-bearing linear stage (ABL10100-LN) is customized with a 3-axis optical fiber positioner holder and provides a scanning direction parallel to the fiber axis. The optical fiber without coating is introduced at the V-

groove and held by two magnetic clamps. 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 are possible due to a load cell (Interface SML series) previously calibrated. For writing 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). This simple arrangement allows the automated writing of gratings by simply translating the optical fiber at constant velocity along the longitudinal direction (X), with the signal modulation turned on. The LPFG was fabricated with a 140 nJ pulse energy, 50 μ m/s scan velocity, 4 cm of length, a refractive index modulation period of 192 μ m, 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 to 1700 nm with 10 nm resolution.

The TiO₂ thin-film deposition around the LPFG was realized by thermal evaporation of pure titanium in a controlled oxygen atmosphere using an electron beam evaporator Auto 306 (Edwards Ltd, U.K.) [8]. Consequently, the optical fiber was assembled in a rotary system, coupled to the vacuum chamber, to produce a homogeneous thin film thickness of 20 nm with a rotation velocity of 5 rpm. The optical fiber deposition process was calibrated with a quartz microbalance model FTM5 (Edwards Ltd., U.K.), which provides the value of the film thickness deposited on a flat substrate [8].

Two characterization setups were developed to test the LPFG before and after the deposition of the thin film in SRI and temperature measurements. The SRI characterization was performed with water with different salt concentrations whose refractive indexes were measured at 589.3 nm with an Abbe refractometer (Atago, DR-A1) and varied from 1.333 to 1.371. The grating region was introduced at the glass V-groove, parallel aligned to the fiber axis, and previously prepared with the solution to be tested. After each measurement, both optical fiber and the V-groove were cleaned with acetone to avoid contamination to the next solution. The temperature characterization setup is composed of a recipient prepared with a V-groove with two magnetic clamps to hold the optical fiber straight. The recipient was filled with water at 100°C, and the transmitted signal was measured during water cooling to room temperature. The temperature was measured with a type K thermocouple thermometer (AMECaL ST-9612).

In both characterization methods, the optical fiber was stretched with an axial tension produced by a weight of 5.8 g. The broad spectrum of a halogen lamp signal transmitted in the grating was measured with an optical spectrum analyzer (OSA, ANDO AQ-6315B) without polarization control from 1200to 1700 nm with a 2 nm resolution. The described processes are illustrated in Fig. 1.



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

3. Experimental results and discussion

The LPFG operating near its phase matching turning point, fabricated with a RI modulation period of $192 \,\mu$ m, yielding a dual resonance wavelength at ~1450 nm, associated with the LP_{1,12} asymmetric cladding mode. To assess the SRI sensitivity, the TAP LPFG bare and coated with 20 nm of TiO₂ were exposed to different SRI, and the results presented in Fig. 2.



Fig. 2. Uncoated TAP LPFG: (a) Transmission spectra for several values of the SRI, (b) Normalized wavelength shift as a function of the SRI, and (c) Normalized wavelength shift as a function of time when the surrounding medium undertakes step variations; TAP LPFG coated with 20 nm thick TiO₂ film: (d) Transmission spectra for several values of the SRI, (e) Normalized wavelength shift as a function of the SRI, and (f) Normalized wavelength shift as a function of time when the surrounding medium undertakes step variations.

The TAP LPFG transmission spectra presented in Fig 2 (a) and (d) exhibits two peaks in the air (RI = 1.000), and two separated peaks with increasing bifurcation as the SRI increases. The homogeneous thin film thickness of 20 nm deposited around the grating increases the two peaks separation, reaching a separation of 18.13 nm (blue shift - 8.88 nm, and red shift +9.25 nm). In addition, the intensity of both resonances diminishes. It was verified that the TiO₂ thin film increases the wavelength sensitivity of the TAP LPFG to changes in the SRI for values lower than the cladding RI. The normalized wavelength shift as a function of the SRI, from 1.333 to 1.371, is shown in Fig 2 (b) and (e) for the uncoated and coated TAP LPFG, respectively. The uncoated TAP LPFG presents a wavelength sensitivity of -449.62, and 668.96 nm/RIU (RIU – RI Unit) for the resonances λ_1 and λ_2 , respectively. Conversely, the coated TAP LPFG offers an improvement of the wavelength sensitivity to -532.76 nm/RIU, and 858.21 nm/RIU for the resonances λ_1 and λ_2 , respectively. The RI spectral resolution, R_{RI} was also estimated based on the step changes and associated measurement fluctuations, through the equation, $R_{RI} = (\Delta RI/\Delta \lambda)\sigma_{Max}$, where ΔRI is the RI variation, $\Delta \lambda$ is the variation in the wavelength of the resonant band and, σ_{Max} is the highest standard error deviation associated with the step variation of the external RI [9]. The normalized wavelength shift of the resonance λ_2 as a function of time when the surrounding medium undertakes step variations is shown in Fig. 2. (c) and (f) for the uncoated and coated TAP LPFG, respectively. Refractive index resolutions of 2.31×10^{-4} , and 3.63×10^{-4} were achieved for the resonances λ_1 and 1.78×10^{-4} , and 1.52×10^{-4} , for the resonances λ_2 of the uncoated and coated TAP LPFG, respectively.

To assess the temperature sensitivity, the TAP LPFG bare and coated with 20 nm of TiO_2 were immersed in water at different temperatures, and the results presented in Fig. 3.



Fig. 3. Transmission spectra of TAP LPFG (a) uncoated, (b) coated with 20 nm thick TiO₂ film for several values of the SRI and, (c) normalized wavelength shift as a function of the water temperature.

The TAP LPFG transmissions spectra presented in Fig. 3 (a) and (b) exhibit two resonances separated by 31.35 nm (blue shift -15.83 nm, and red shift +15.52 nm) and 12.63 nm (blue shift -6.75 nm, and red shift +5.88 nm), repectively, at a water cooling temperature range from 80 to 20 °C. The temperature dependence of the water RI explains the increase of the two resonances wavelength separation with the decrease of the water temperature. The normalized wavelength shift to the initial state (at 80 °C) as a function of the water temperature, from 80 to 30 °C, is shown in Fig 3 (C) for the uncoated and coated TAP LPFG. The uncoated TAP LPFG presents a wavelength sensitivity to temperature variation of -0.320 and 0.314 nm/°C for the resonances λ_1 and λ_2 , respectively. However, the wavelength sensitivity of the coated TAP LPFG falls to -0.132, and 0.113 nm/ °C for the resonances λ_1 and λ_2 , respectively. This sensitivity decrease is not unexpected because of the considerably reduced sensitivity for wavelengths away from the TAP [3].

4. Conclusion

A TAP LPFG in an SMF-28e through fs-laser direct writing was fabricated and coated with 20 nm of TiO₂ thin film. The LPFG operating near its phase matching turning point was fabricated with a refractive index modulation period of 192 μ m, yielding a dual resonance wavelength at ~1450 nm, associated with the LP_{1,12} asymmetric cladding mode. It was verified that the TiO₂ thin film increases the wavelength sensitivity of the TAP LPFG to changes in the SRI for values lower than the cladding RI. Sensitivities of -532.76nm/RIU, and 858.21 nm/RIU for the coated TAP LPFG resonances λ_1 and λ_2 , were achieved. The two resonances different response is related to the non-symmetrical phase matching curves of the cladding mode, which has a λ^4 dependence [3]. The sensitivity of the fabricated LPFGs can be optimized by appropriate choice of the thickness of the TiO₂ thin film and period of the grating to ensure that the LPFG operates at both the mode transition region and the phase matching turning point. The relatively low complexity and the robust nature of the fabricated device open the possibility to chemical and bio-chemical sensing in practical applications.

5. Acknowledgments and funding

This activity is supported by grant, SFRH/BD/110035/2015, from the Ministry of Education and Science of the Portuguese Government. The research leading to these results was developed in the framework of Project SAFE WATER - "On Chip Whispering Gallery Mode Optical Microcavities For Emerging Microcontaminant Determination In Waters", supported and co-funded by the European Commission, Directorate-General Communications Networks, Content and Technology (DG CONNECT) under the ERA-NET Cofund scheme - Horizon 2020 "Horizon 2020 – the Framework Programme for Research and Innovation (2014-2020)".

6. References

- [1] S. Korposh, S. James, R. Tatam, and S.-W. Lee, "Optical fibre long-period gratings functionalised with nano-assembled thin films: Approaches to chemical sensing," *Curr. Trends Short-and Long-period Fiber Gratings*, p. 63, 2013.
- [2] R. Y. Wong, D. Hu, J. Juan, and M. Ibsen, "Applications of Optical Fibers for Sensing," in IntechOpen, 2019.
- [3] X. Shu, L. Zhang, and I. Bennion, "Sensitivity characteristics of long-period fiber gratings," J. Light. Technol., vol. 20, no. 2, pp. 255– 266, 2002.
- [4] P. Biswas, N. Basumallick, S. S. Bandyopadhyay, K. Dasgupta, A. Ghosh, and S. S. Bandyopadhyay, "Sensitivity enhancement of turnaround-point long period gratings by tuning initial coupling condition," *IEEE Sens. J.*, vol. 15, no. 2, pp. 1240–1245, 2014.
- [5] X. Lan, Q. Han, T. Wei, J. Huang, H. Xiao, and S. Member, "Turn-Around-Point Long-Period Fiber Gratings Fabricated by CO Laser Point-by-Point Irradiations," *IEEE Photonics Technol. Lett.*, vol. 23, no. 22, pp. 1664–1666, 2011.
- [6] M. Gambhir and S. Gupta, "Review of turn around point long period fiber gratings," J. Sens. Technol., vol. 5, no. 04, p. 81, 2015.
- [7] D. Viveiros *et al.*, "Spectral Tuning of Long Period Fiber Gratings Fabricated by Femtosecond Laser Micromachining through Thermal Annealing," in *Multidisciplinary Digital Publishing Institute Proceedings*, 2019, vol. 15, no. 1, p. 4.
- [8] L. Coelho, D. Viegas, J. L. Santos, and J. M. M. De Almeida, "Sensors and Actuators B : Chemical Enhanced refractive index sensing characteristics of optical fibre long period grating coated with titanium dioxide thin films," *Sensors Actuators B. Chem.*, vol. 202, pp. 929–934, 2014.
- [9] L. Coelho et al., "Aptamer-based fiber sensor for thrombin detection," J. Biomed. Opt., vol. 21, no. 8, p. 87005, 2016.