Enhancing sensitivity of long-period gratings by combined fiber etching and diamond-like carbon nano-overlay deposition

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ABSTRACT

This work presents an application of reactive ion etching (RIE) followed by diamond-like carbon (DLC) nano-overlay deposition using radio frequency plasma enhanced chemical vapor deposition (RF PECVD) method for effective tuning of the refractive-index (RI) sensitivity of long-period gratings (LPGs). Both etching and deposition take place within one process. Combination of both plasma-based processes allows for well controlled tuning of the LPG sensorial response up to its operation at both dispersion turning point (DTP) of higher order cladding modes and mode transition regime. As a result of processing, RI sensitivity can be enhanced up to over 12,000 nm/RIU per single resonance in narrow RI range (1.3344-1.3355) and over 2000 nm/RIU in broader RI range (1.34-1.356).

Keywords: optical fiber sensors, long-period gratings, refractive index sensing, thin films, diamond-like carbon, chemical vapor deposition, reactive ion etching

1. INTRODUCTION

Long-period gratings (LPGs) have been known for over a decade. LPGs are a periodic modulation of the refractive index along the length of an optical fiber. Under special phase-matching conditions, the grating couples the fundamental core mode to discrete cladding modes that are attenuated due to absorption and scattering. The coupling is wavelength-dependent, so a spectrally selective loss can be obtained. A number of sensors based on the LPGs have been proposed for temperature, strain, hydrostatic pressure, bending and refractive index sensing [e.g., 1, 2].

LPGs offers the highest sensitivity to a number of measurands at vicinity of dispersion turning point (DTP) of higher order cladding modes [1]. At this working point double resonances effect can be observed in LPG transmission spectrum and the resonances shift contradictory to each other under certain external influence. The LPG at DTP offers the highest sensitivity, but the resonances there are relatively broad and shallow, which makes it difficult to apply them in sensorial interrogation. Moreover, the range of the highest sensitivity is limited to DTP and drops significantly away from it [3].

It has been also shown that the deposition of some high-refractive-index (high-n) nano-coatings significantly modifies the sensitivity of the LPG structures to variations of external refractive index (n_{ext}) [4]. Such coatings make it possible to optimize the interactions of the light guided in the fiber and in the coating, thus tuning the intrinsic sensitivity of optical fiber devices to a certain n_{ext}. A high sensitivity in the specified range of n_{ext} can be achieved by precise adjustment of the thickness and the optical properties of the coatings. At such conditions one of the modes start to propagate in the overlay and induces transition of other modes.

Attempts of both effects have been reported, i.e., DTP and mode transition (MT), for enhancing LPG sensing properties [5, 6]. It must be noted that combining the effects is not trivial due to the fact, that reaching DTP typically requires precise etching of the fiber cladding and consecutive overlay deposition tunes the grating away from the DTP. That is why the LPG must be over-etched and then coated with high-n overlay in order to reach both the DTP and MT effects. Up to date, the etching and deposition were done separately using HF acid etching and liquid [5] or vapor-based precursor deposition [6].

In this work, we discuss possibility of reaching DTP and MT conditions using plasma-based methods. The methods, namely reactive ion etching (RIE) and radio-frequency plasma-enhanced chemical vapor deposition (RF PECVD) have shown capability for application on LPGs [7, 8]. They both allow for precise control of the etched cladding and deposited film thickness in nanometer range. Moreover, obtained with RF PECVD method diamond-like carbon (DLC) thin films

24th International Conference on Optical Fibre Sensors, edited by Hypolito José Kalinowski,
José Luís Fabris, Wojtek J. Bock, Proc. of SPIE Vol. 9634, 963456 · © 2015 SPIE
CCC code: 0277-786X/15/$18 · doi: 10.1117/12.2195214

are known for their high mechanical and chemical resistance [9], what make them promising for application in long-term sensing. The influence of both etching and DLC-overlay deposition processes on RI sensitivity of the obtained sensors is discussed.

2. EXPERIMENTAL DETAILS

The long period gratings were written in hydrogen-loaded standard Corning SMF28 fiber using Pulse Master 840 high-power KrF excimer laser (λ = 248 nm). A pulse repetition rate was set to 100 Hz, pulse duration to 12 ns, and peak pulse energy was about 10 mJ. The UV exposure has been done through an amplitude chromium mask (Λ = 226.8 µm) for about 7 minutes. In order to stabilize the LPGs optical properties, they were annealed after exposure at 150 °C for approx. 4 h to release the excess of hydrogen. In order to enhance the RI sensitivity towards DTP, the gratings were etched in HF acid for about 3 h. The etching procedure resulted in the sensor operation at the vicinity of the dual resonance regime (DTP). The sensors are 4 cm long, and the sensitivity of the structures close to DTP was approx. 3000 nm/RIU, e.g., [3].

The Oxford PlasmaPro NGP80 system was used for the etching of fiber cladding and deposition of DLC thin films on the LPGs and on the reference oxidized silicon (SiO_2) wafers. The LPG samples were cleaned with isopropanol before placing them in the plasma reactor. Then, the LPG samples were placed on U-type holder 7.2 mm over the electrode and the reference Si wafers were placed next to them on the holder [10]. The RF PECVD process took 4 to 12 minutes and was conducted with CH_4 flow of 50 sccm, pressure 30 mTorr, power 150 W and temperature 20 °C. Next, the RIE process aiming for DLC and cladding etching was performed according to data given in [10] and [7], respectively.

The optical transmission of the LPG in the range of λ = 1550-1750 nm was monitored using a NKT Photonics SuperK COMPACT supercontinuum white light laser source and Yokogawa AQ6375 optical spectrum analyzer. The ambient temperature (T) during the measurements was set to 22 °C and monitored with HP 34970A Data Acquisition Unit equipped with a thermocouple. The RI sensitivity of the LPGs has been measured for samples immersed in glycerin/water mixtures with n_D from 1.33 to 1.45 RIU [3]. The LPGs were kept under the same tension during all the experiments.

3. RESULTS AND DISCUSSION

The aim of this work is to obtain LPGs working both at DTP and MT. One of common methods for obtaining DTP is application of precise etching of optical fiber cladding [3, 7]. Since fabricated and HF-etched LPGs show DTP when they are surrounded by air and work away from this point, i.e., resonances are well spectrally separated when are immersed in water, we used RIE for precise tuning of the fiber cladding. Response of the LPG to etching is shown in Figure 1. After in total 21.5 minutes of etching with RF power of 100 W, we reached DTP for LPG immersed in water (n_D=1.3330 RIU). It can be seen in Figure 1, that longer etching induces merging of the resonances at $\lambda \approx$ 1630 nm and the shift in resonance wavelength is no longer possible when water surrounds the LPG. Keeping in mind that deposition of high-*n* nano-overlay such as DLC will induce separation of the resonances, we intentionally over-etched fiber's cladding. As a result of this process the LPG experienced DTP for n_{ext} over 1.36 RIU.

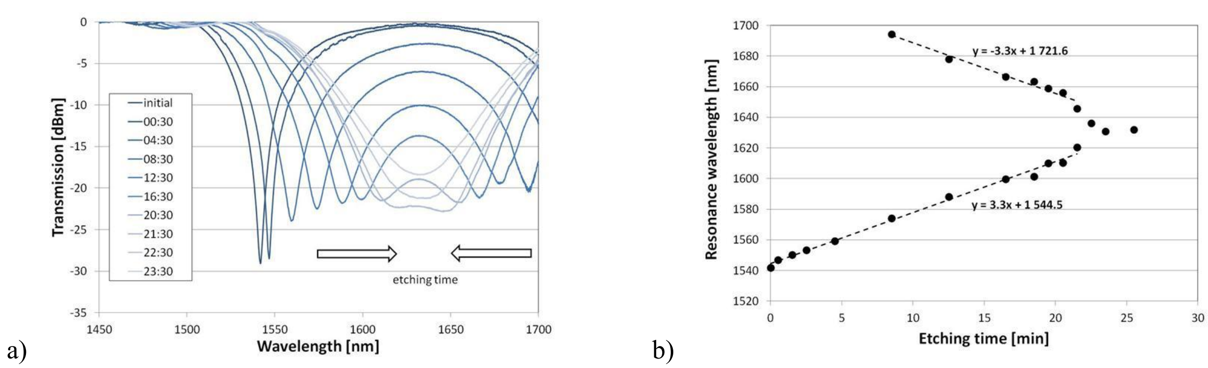
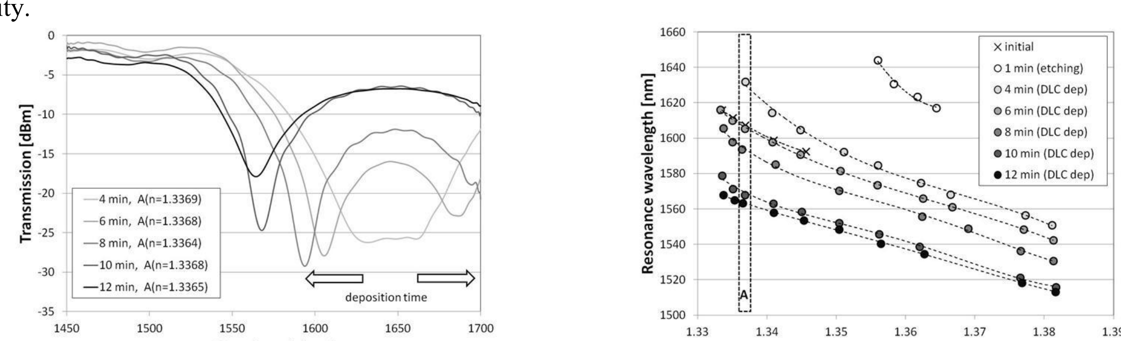


Fig. 1. Effect of RIE time on (a) spectrum of the LPG and (b) shift of the resonance wavelength. The measurements were performed when the LPG was immersed in water (n_D=1.3330 RIU).

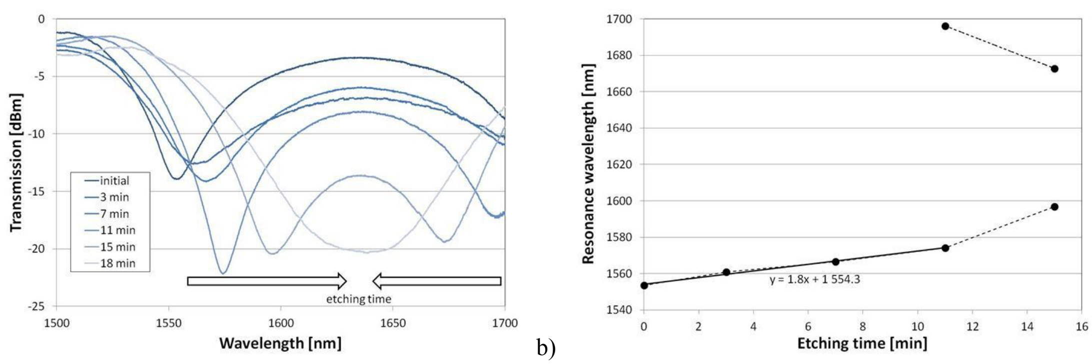
Effect of DLC deposition on LPG response to n_{ext} is shown in Figure 2. Spectral separation between resonances increases with duration of the deposition process. When the process is 4 min.-long, DTP is reached again when n_{ext} is slightly higher than the one for water. High sensitivity in this range is highly desired when the LPG is planned to be used for label-free sensing [3]. Longer deposition time induces further separation between the resonances. As can be seen in Figure 2b, the sensitivity in this n_{ext} range is high for sample with DLC deposited in 4 min-long process. The sensitivity in this range is highly influenced by the DTP effect. For longer deposition time, i.e. 6 minutes, the sensitivity drops and again increases for 8 and 10 min.-long deposition. The second increase in sensitivity is in turn influenced by MT effect. When results obtained for the same spectral range are compared, i.e., initial response of LPG and the response of the sample coated in 4 min.-long process, it can be seen, that even with thinner film than required for MT the sensitivity is slightly improved. The results indicate importance of obtaining both effects at the same time for improvement of RI sensitivity.



a) b)

Fig. 2. Response to external refractive index for the LPG with DLC deposited in processes lasting from 4 to 12 minutes, where (a) shows spectra at selected $n_{ext} \approx 1.3368$ RIU and (b) shows resonance wavelength shift. Response of the LPG before (initial) and after 1 min-long RIE is given for comparison.

Since 8 min.-long DLC deposition allows for obtaining MT conditions, we employed RIE followed by DLC deposition with constant time. It can be seen in Figure 3, that for 8 min.-long deposition it is required to etch the fiber cladding for another 18 min. in order to reach conditions close to the DTP. The resonance wavelength shift induced by etching and deposition is about 1.8 nm/min, and it is about twice less effective than for LPG with no coating (Figure 1b).



a) b)

Fig. 3. Evolution of the LPG response with RIE of fiber cladding and subsequent 8 min.-long DLC film deposition process, where (a) shows spectra of the LPG after each process set and (b) resonance wavelength shift induced by the processes. The measurements have been taken when LPG was immersed in water (n_D=1.3330 RIU).

Finally, effect of combined RIE and DLC deposition were compared for different etching time (Figure 4). Etching of the fiber cladding resulting in tuning the LPG towards DTP significantly improves the sensitivity. In range where the sensitivity is close to linear (n_{ext} from about 1.34 to 1.356 RIU), we have improved it by almost 30 %. In range where DTP significantly influences the sensitivity (n_{ext} = 1.3344-1.3355 RIU), it has been improved up to 12,360 and -10,090 nm/RIU for red and blue shifting resonances, respectively. In case of the LPG with no coating in the same RI range the sensitivity reaches 4,375 nm/RIU.

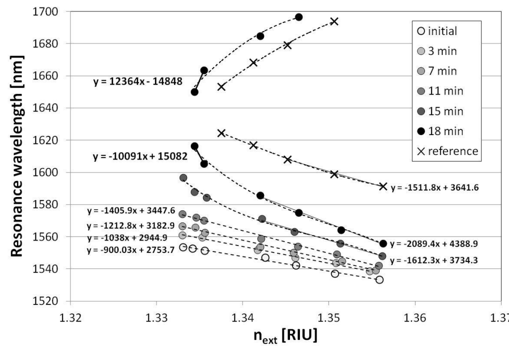


Fig. 4. Resonance wavelength shift induced by n_{ext} for different fiber cladding RIE time. Each RIE process was followed by 8 min-long DLC deposition. Results for the LPG working in DTP with no etching and no coating is shown for comparison. The sensitivity is given in range 1.34 to 1.356 RIU.

4. CONCLUSIONS

In this work, we applied both effects, i.e. dispersion turning point and mode transition, for enhancing refractive index sensing properties of LPGs. The effects have been applied by combining reactive ion etching and radio-frequency plasma enhanced chemical vapor deposition of diamond-like carbon. Both methods can be applied in the same plasma reactor; it makes the LPG processing fully automated and very precise. We have compared effects associated to the applied processes separately and as a set. According to our best knowledge, the obtained sensitivity in narrow RI range exceeding 12,000 nm/RIU for a single resonance is the highest reported up to date for LPGs. The high sensitivity for liquid solutions with near water RI allows for treating the nano-coated LPG as an attractive platform for label-free biosensing. Moreover, we have also enhanced sensitivity by almost 30 % in broader RI range, which makes the sensor more attractive for RI monitoring in various applications, e.g., in industrial conditions.

ACKNOWLEDGEMENTS

The authors gratefully acknowledge support for this work from the Natural Sciences and Engineering Research Council of Canada, the Canada Research Chairs Program, the National Centre for Research and Development of Poland within the LIDER program, and the Polish National Science Centre within 2011/03/D/NZ7/02054 project.

REFERENCES

[1] Shu, X., Zhang, L., and Bennion, I., "Sensitivity Characteristics of Long-Period Fiber Gratings," J. Lightwave Technology 20, 255-266 (2002).

[2] Smietana, M., Bock, W.J., Mikulic, P., and Chen, J., "Tuned Pressure Sensitivity of Dual Resonant Long-Period Gratings Written in Boron Co-doped Optical Fiber," Journal of Lightwave Technology 30, 1080-1084 (2012).

[3] Brzozowska, E, Śmietana, M., Koba, M., Górska, S., Pawlik, K., Gamian, A., and Bock, W.J., "Recognition of bacterial lipopolysaccharide using bacteriophage-adhesin-coated long-period gratings," Biosens. Bioelectron. 67, 93-99 (2015).

[4] Del Villar, I., Matias, I., Arregui, F., and Lalanne, P., "Optimization of sensitivity in Long Period Fiber Gratings with overlay deposition," Opt. Express 13, 56-69 (2005).

[5] Pilla, P., Trono, C., Baldini, F., Chiavaioli, F., Giordano, M., and Cusano, A., "Giant sensitivity of long period gratings in transition mode near the dispersion turning point: an integrated design approach," Opt. Lett. 37(19), 4152-4154 (2012).

[6] Smietana, M., Koba, M., Brzozowska, E., Krogulski, K, Nakonieczny, J., Wachnicki, L., Mikulic, P., Godlewski, M., and Bock, W.J. "Label-free sensitivity of long-period gratings enhanced by atomic layer deposited TiO2 nano-overlays," Opt. Express (2015) - in press.

[7] Śmietana, M., Koba, M., Mikulic, P., Boc,k W.J., "Measurements of reactive ion etching process effect using long-period fiber gratings," Opt. Express 22, 5986-5994 (2014).

[8] Smietana, M., Szmidt, J., Korwin-Pawlowski, M.L., Bock, W.J., and Grabarczyk, J., "Application of diamond-like carbon films in optical fibre sensors based on long-period gratings," Diam. Relat. Mater. 16, 1374-1377 (2007).

[9] Robertson, J., "Diamond-like amorphous carbon," Materials Science and Engineering R37, 129-281 (2001).

[10] Śmietana, M., Koba, M., Mikulic, P., Bock, W.J., "Tuning properties of long-period gratings by plasma post-processing of their diamond-like carbon nano-overlays," Meas. Sci. Technol. 25, 114001 (2014).