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Combined Plasma-Based Fiber Etching and Diamond-Like Carbon Nano-overlay Deposition for Enhancing Sensitivity of Long-Period Gratings

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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 of higher order cladding modes and mode transition regime. As a result of the processing, the RI sensitivity has been enhanced up to over 12,000 nm/RIU per single resonance in narrow RI range (1.3344-1.3355 RIU) and over 2,000 nm/RIU in broader RI range (1.34-1.356 RIU). Experimental results has been supported by numerical analysis that show capabilities for further significant improvement of both RI sensitivity and range of RI measured with the enhanced sensitivity.**

***Index Terms*— optical fiber sensors, long-period gratings, refractive index sensing, plasma-based processing, thin films, diamond-like carbon, chemical vapor deposition, reactive ion etching.**

I. 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 [1]. 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 has been proposed for temperature, strain, hydrostatic pressure, bending and refractive index (RI) sensing [e.g., 2, 3]. It has also been shown that high RI sensitivity can be successfully applied other purposes including label-free biosensing [4, 5].

LPGs offer the highest sensitivity to a number of measurands at vicinity of dispersion turning point (DTP) of higher order cladding modes [2]. 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 [5]. This property of LPG makes high accuracy measurements of RI in broad range difficult.

It has also been 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 RI (n_{ext}) [6]. Such coatings make it possible to optimize the interactions of 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, i.e., DTP and mode transition (MT), have been reported for improving LPG sensing properties [7, 8]. Recently, it has been also shown as a result of numerical analysis, that this approach allows for obtaining sensitivities reaching 143,000 nm/RIU in narrow RI range and for fiber diameter reduced to 34.8 μm [9]. However, it must be noted that combining the effects is not trivial in practice 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 wet etching in hydrofluoric (HF) acid and liquid [7] or vapor-based [8, 10] precursor deposition.

In this work, we discuss possibility of reaching DTP and MT conditions using only plasma-based methods [11]. The methods, namely reactive ion etching (RIE) and radio-frequency plasma-enhanced chemical vapor deposition (RF PECVD) have shown capability for application on LPGs [12,

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13]. 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 are known for their high mechanical and chemical resistance [14], what makes 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. We support experimental data with numerical analysis, which show unquestionable potential of the approach for both improving RI sensitivity and RI range where high sensitivity can be reached.

II. 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 pre-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) of LP_{09} cladding mode observed for LPG surrounded by air [10]. The sensors are 4 cm long, and the sensitivity of the structures close to DTP was approx. 3,000 nm/RIU, e.g., [5].

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 [12]. The LPG samples were cleaned with isopropanol before placing them in the plasma reactor. Then, the LPG samples were placed on U-type holder at 7.2 mm over the electrode and the reference Si wafers were placed next to them on the holder [15]. The distance between LPG and the electrode has been adjusted for symmetrical fiber surface processing [16]. 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. The RIE process aiming for DLC and cladding etching was performed according to data given in [16] and [12], 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 AQ6370C 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 1.333 to 1.385 RIU [5]. The LPGs were kept under constant tension during all the experiments.

The experimental stages were following: (1) plasma-based etching of fiber cladding up to DTP observed after LPG immersion in water, (2) DLC deposition of nano-overlays with different thickness for selection of the thickness for which MT takes place, (3) etching of the cladding followed by DLC deposition with properties selected in the previous stage, for reaching DTP again. Effects of the processes were each time monitored for LPG immersed in various n_{ext}.

III. Results and Discussion

There is a number of influences that can shift the resonance wavelengths (λ_{res}^m) of the LPG [1-3]. The main relation describing wavelength-dependent coupling from the guided core mode (LP_{01}) to the m[th] cladding mode (LP_{0m}) is shown in (1), where (n_{eff}^{01}) is the effective refractive index of the propagating core mode, (n_{eff}^{0m}) is the effective refractive index of the m[th] cladding mode and (Λ) is the period of the LPG.

$$\lambda_{res}^m = \left(n_{eff}^{01} - n_{eff}^{0m}\right)\Lambda \quad (1)$$

A resonance wavelength shift can be induced by variation of either the period of the grating or the effective indices of the modes. As far as the influence of external RI is concerned, only n_{eff}^{0m} can be influenced by external medium, which in specific case can be a bio-overlay. According to (1) when the external RI increases or when the overlay is formed on the LPG surface, the λ_{res}^m shifts towards shorter wavelengths (experiences a blue shift). When the LPG works in vicinity of DTP the resonances coming from the same cladding mode shift towards opposite directions, e.g., for increase in n_{ext} the resonance at lower and higher wavelength experience blue and red shift, respectively.

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 [5, 12]. Since our fabricated and HF-etched LPGs show DTP of LP_{09} cladding mode 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. Response to n_{ext} for LPG etched up to DTP in water and after 1 min.-long over etching is shown for comparison in Figure 2b.

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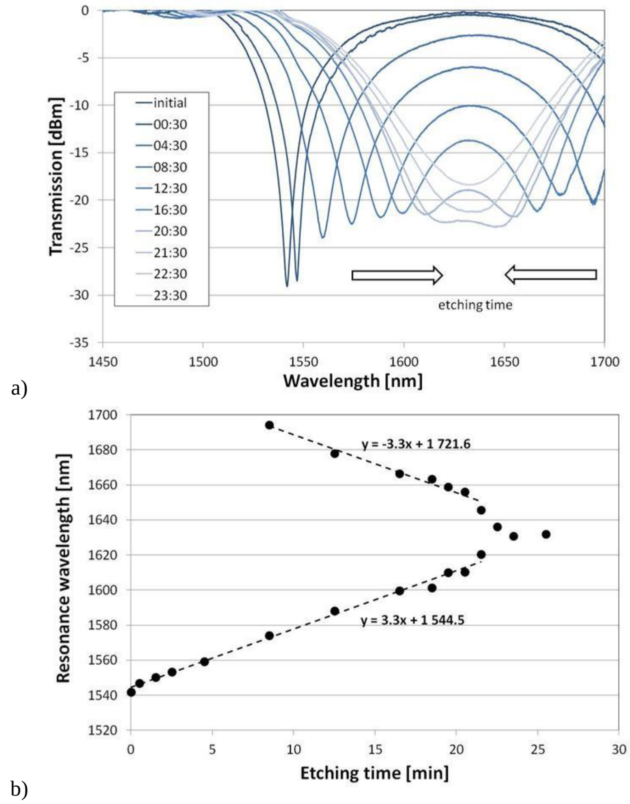

Fig. 1. Effect of RIE time on (a) spectrum of the LPG and (b) shift of the resonance wavelength. The measurements were performed when the LPG was immersed in water (n_D=1.3330 RIU).

Second stage of the experiment was performed next. Effect of DLC deposition on LPG response to n_{ext} is shown in Figure 2. Spectral separation between resonances increases with duration of the DLC deposition process. When the process is 4 min.-long, DTP is reached again when n_{ext} is slightly higher than the one of water. High sensitivity in this range is highly desired when the LPG is planned to be used for label-free sensing [5, 8]. 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 overlay deposited in 4 min-long process. The sensitivity in this range is highly influenced by the DTP effect. For longer deposition time, i.e., longer than 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.

Since 8 min.-long DLC deposition allows for obtaining MT conditions, in third stage of the experiment we employed RIE followed by DLC deposition, the later with the constant time. It can be seen in Figure 3, that 8 min.-long deposition requires the fiber cladding to be etched 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). Finally, effect of combined RIE and DLC

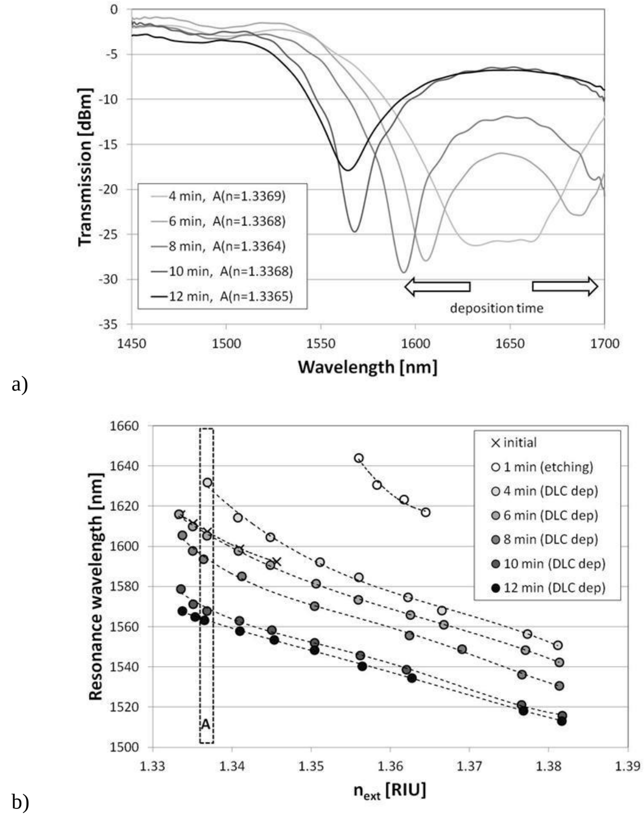

Fig. 2. Response to external refractive index for the LPG with DLC deposited in processes lasting from 4 to 12 minutes, where (a) shows spectra at selected $n_{ext} \approx 1.3368$ RIU and (b) shows resonance wavelength shift. Response of the LPG before (initial) and after 1 min-long RIE is given for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

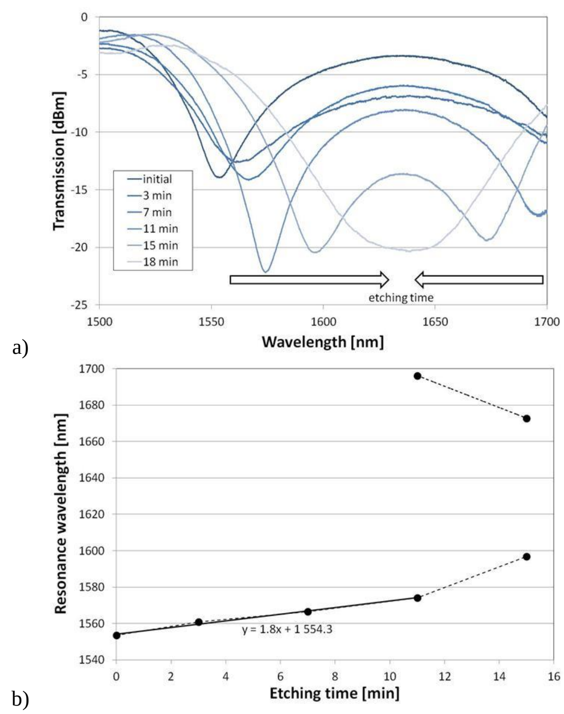

Fig. 3. Evolution of the LPG response with RIE of fiber cladding and subsequent 8 min.-long DLC film deposition process, where (a) shows spectra of the LPG after each process set and (b) resonance wavelength shift induced by the processes. The measurements have been taken when LPG was immersed in water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

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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.

Taking into consideration huge capabilities offered by the combined approach, we performed numerical simulations of the investigated case. The results of numerical analysis of the LPG response to etching and overlay formation are shown in Figure 5. We assumed DLC overlay with n = 2 RIU and its thickness in range of up to 60 nm [15]. For each thickness the diameter of the fiber cladding has been reduced down to obtain resonances with spectral distance of 50 nm. It can be seen that for thicker DLC films significant reduction in fiber cladding radius is needed. The relation between overlay thickness and the reduced cladding radius is exponential (Figure 5a). Moreover, the combined processes result in shifting of the DTP towards lower wavelength. The same effect has been observed experimentally (Fig. 4). For deep reduction of the cladding radius (840 nm) followed by deposition of 50 nm overlay it can be possible to take advantage of both DTP and MT effects, resulting in significantly enhanced sensitivity up to 5,600 nm/RIU in relatively broad RI range (n_{ext} from 1.33 to 1.37 RIU) for a single resonance. Deeper etching and deposition of the thicker overlay can enhance the sensitivity even more, but this in turn narrows high-sensitivity-range down to 1.33-1.34 RIU. The numerical analysis confirm trends observed experimentally. For further sensitivity enhancement both the combined processes need to be longer or carried more efficiently.

IV. 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 exceeding 12,000 nm/RIU for a single resonance and over 22,000 nm/RIU for a spectral difference between the resonances at DTP in narrow RI range close to n_D = 1.3330 RIU are the highest reported experimentally up to date for LPGs. Previously reported sensitivity of 9,100 nm/RIU was reached for higher RI range and for not as robust as DLC nano-coatings [7]. 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. It has been also shown as a result of numerical analysis, that the sensitivity can be further enhanced by longer or more effective processing, i.e., carried with a higher power. However, the increase in sensitivity is followed by narrowing high-sensitivity range. These effects must be taken into consideration when certain sensing applications are foreseen.

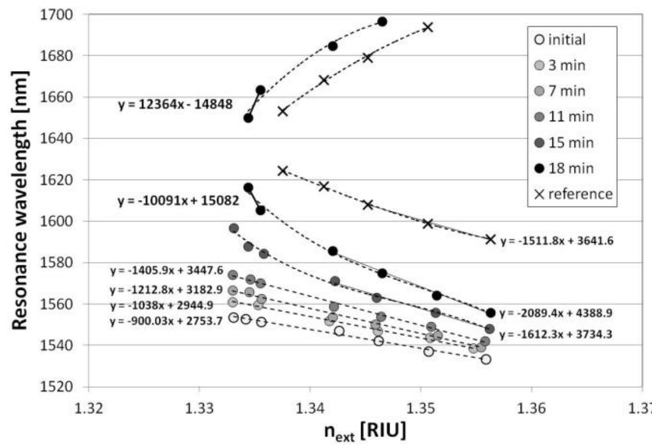
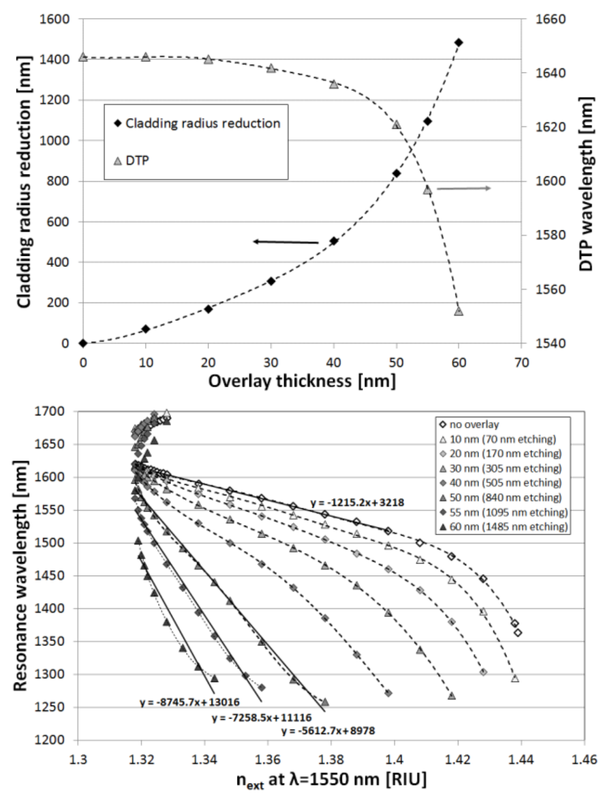


Fig. 4. Resonance wavelength shift induced by n_{ext} for different fiber cladding RIE time. Each RIE process was followed by 8 min-long DLC deposition. Results for the LPG working in DTP with no etching and no coating is shown for comparison. The sensitivity is given in range 1.34 to 1.356 RIU.

a)



b)

Fig. 5. Simulation of the LPG response to combined etching and overlay deposition, where (a) shows DTP wavelength shift for both cladding reduction and increase in overlay thickness and (b) shows resonance wavelength shift induced by change in n_{ext} for each overlay thickness and cladding diameter reduction. Assumed conditions: overlay n = 2 RIU, initial spectral distance between the resonances is 50 nm.

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