

Towards refractive index sensitivity of long-period gratings at level of tens of μm per refractive index unit: fiber cladding etching and nano-coating deposition

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Abstract: In this work we report experimental results on optimizing the refractive index (RI) sensitivity of long-period gratings (LPGs) by fiber cladding etching and thin aluminum oxide (Al_2O_3) overlay deposition. The presented LPG takes advantage of work in the dispersion turning point (DTP) regime as well as the mode transition (MT) effect for higher-order cladding modes (LP_{09} and LP_{010}). The MT was obtained by depositing Al_2O_3 overlays with single-nanometer precision using the Atomic Layer Deposition method (ALD). Etching of both the overlay and the fiber cladding was performed using hydrofluoric acid (HF). For shallow etching of the cladding, i.e., DTP observed at $n_{\text{ext}} = 1.429$ and 1.439 RIU for an LPG with no overlay, followed by deposition of an overlay of up to 167 nm in thickness, HF etching allowed for post-deposition fine-tuning of the overlay thickness resulting in a significant increase in RI sensitivity mainly at the DTP of the LP_{09} cladding mode. However, at an external RI (n_{ext}) above 1.39 RIU, the DTP of LP_{010} was noticed, and its RI sensitivity exceeded 9,000 nm/RIU. Deeper etching of the cladding, i.e., DTP observed for n_{ext} above 1.45 RIU, followed by the deposition of thicker overlays (up to 201 nm in thickness) allowed the sensitivity to reach values of over 40,000 nm/RIU in a narrow RI range. Sensitivity exceeding 20,000 nm/RIU was obtained in an RI range suitable for label-free biosensing applications.

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1. Introduction

Optical devices offering high refractive index (RI) sensitivity not only are capable of detecting contamination or aging of liquids [1], but also can be applied to detect differences in the thickness or the optical properties of a material formed on their surface. Label-free biosensors [2] and chemical sensors [3] have been presented, based on high RI sensitivity to changes in optical properties at the optical fiber's surface. Long-period gratings (LPGs) are considered to be very promising RI sensing devices [4]. The LPG is a periodical modulation of the refractive index in the core of single-mode optical fiber, which allows for coupling between the fundamental core mode (LP₀₁) and *m* cladding modes (LP_{0m}). The coupling is observed as a series of resonances in the transmission spectrum of the LPG. Since the coupling depends on the propagation conditions in the core and cladding modes, namely their effective refractive indices (n_{eff}), and on the grating period (A), a spectral shift of the resonances resulting from changes of n_{eff} or A can be used for sensing purposes. The RI sensitivity comes from variation in the n_{eff} of the cladding modes induced by change in the external RI (n_{ext}) and is typically defined as a shift of resonance wavelength (λ^m) per RI unit (RIU) [5].

Etching of the optical fiber cladding and high-refractive-index (high-*n*) thin-overlay deposition are typical methods for enhancing the RI sensitivity of optical fiber sensors, including those based on LPGs. Application of various liquid-based techniques (e.g., immersion in HF acid [6]) or vapor-based methods (reactive ion etching in SF₆ gas [7]) for fiber surface modification have been suggested by various research groups. The etching process typically makes it possible to tune the properties of the grating up to the dispersion turning point (DTP), where LPGs show the highest RI sensitivity reaching 2,600 or even

6,900 nm/RIU in a very narrow RI range [8]. It has also been shown that when a high- n thin-film overlay is deposited on the surface of an LPG, its RI sensitivity can be enhanced due to the mode transition (MT) effect [9,10]. For certain overlay properties and range of n_{ext} , the overlay can play a role of a waveguide and the lower order cladding modes are gradually transitioned from the LPG to the overlay. The transition is responsible for redistribution in n_{eff} of the cladding modes. That is why to reach MT effect it is required to precisely determine properties of the overlay. Control of the overlay thickness to the nm level has been proposed through various liquid-based (Langmuir-Blodgett [9], sol-gel [11], self-assembly monolayers [12]), vapor-based (physical [13] or chemical vapor-based [14]) deposition methods. In recent years, significant attention has been given to application of both processes, i.e., etching and deposition, to obtain extraordinary RI sensitivities in an LPG. Sensitivities reported to date – defined as wavelength shift of a single resonance close to DTP – reach values of 9,100 nm/RIU at $n_{ext} \approx 1.347$ RIU [15] and with a high- n diamond-like carbon overlay, values over 12 000 nm/RIU at $n_{ext} \approx 1.3330$ RIU [16]. It has also been shown theoretically that given a combination of low cladding diameter (34.8 μm) and specific overlay properties (thickness of 313 nm and $n = 1.55$ RIU) the sensitivity can exceed 143,000 nm/RIU for gratings working at an RI close to that of water ($n_{ext} = 1.3330$ RIU) [16]. However, the numerical analysis in that work considered the spectral distance between two resonances at DTP, so for a single resonance the sensitivity will be approximately 50% lower [17]. Another approach, where the LPG structure is formed on the surface of a tapered optical fiber, has been shown to offer sensitivity reaching 168,182 nm/RIU, calculated using the thermo-optic coefficient of measured liquids [18]. Both the sensing schemes require significant reduction of fiber diameter thereby compromising the robustness of the device.

In this work we experimentally explore the possibility of achieving a further increase in LPG sensitivity to n_{ext} by chemical etching of the fiber cladding, followed by well controlled atomic layer deposition (ALD) of aluminum oxide Al_2O_3 nano-overlays [19]. In contrast to theoretical works [17], where fiber diameter was significantly reduced as a means of tracing the coupling of lower order cladding modes, in this work we focused on higher-order cladding modes (LP₀₈-LP₀₁₀) where deep etching of the fiber is not required to trace the resonance [5]. We also investigated the use of HF acid for slight post-deposition etching in the nano-overlay as a means of tuning the LPG sensitivity in the vicinity of $n_{ext} = 1.3330$ RIU where label-free sensors operate.

2. Experimental details

2.1 LPG manufacturing

In this experiment we used commercially available germanium-doped and hydrogen-loaded Corning SMF-28 single-mode optical fiber. We fabricated a set of LPGs by UV irradiation of 4-cm-long fiber section with a KrF excimer laser employing a chromium amplitude mask having $\Lambda = 226.8$ μm . After the UV-writing, the LPGs were annealed in 150 °C for 3 hours in order to release the hydrogen, and thus stabilize the properties of the gratings. After fabrication, the LPGs were immersed in HF acid in order to slightly reduce the diameter of the fiber cladding. During that process, the resonant wavelength was shifted up to DTP. This is a common procedure for maximizing the LPG sensitivity [8].

2.2 Al_2O_3 nano-film deposition, characterization and post-processing

Al_2O_3 thin films were deposited on the LPGs and on reference silicon wafers using the Cambridge NanoTech Savannah S100 system. The deposition procedure was previously reported in [19]. For the Al_2O_3 deposition processes, trimethylaluminum (TMA, $\text{Al}(\text{CH}_3)_3$) and water were used as aluminum and oxygen precursors, respectively. Between gas pulses the chamber was purged with nitrogen. Thickness of the nano-films was controlled by the number of cycles of the ALD process. For the purposes of this experiment, the number of cycles was varied depending on the desired thickness of the layer. The measured deposition rate was approximately 0.1 nm/cycle.

The properties of the Al_2O_3 films deposited on the reference silicon wafers, such as their thickness (d) and n , were determined by a Horiba Jobin-Yvon UVISSEL spectroscopic ellipsometer. The ellipsometric model for Al_2O_3 analysis is described by the Forouhi-Bloomer dispersion formula. According to our previously published results, properties of the films deposited on the fiber and on reference Si wafers in the same process do not differ [19].

Post-deposition reduction in thickness of the overlay was performed by immersion of the nano-coated LPGs in HF acid as for the bare LPGs, but for a shorter period of time.

2.3 LPG measurements

The spectral response of the LPGs was investigated in the wavelength range of 1100 to 1700 nm using a Yokogawa AQ6370C spectrum analyzer and an NKT Photonics SuperK COMPACT supercontinuum white light laser source.

The RI sensitivity was measured by immersing the LPGs in a water-glycerin mixture with n_D ranging from 1.333 to 1.44 RIU. The RI of the liquids was measured using a VEE GEE PDX-95 digital refractometer at a resolution of 10^{-4} RIU. Liquid samples were taken directly from the vicinity of the LPG. Between the immersions, the LPG was rinsed with deionized water. Temperature and strain were kept constant during all the RI measurements.

3. Results and discussion

In order to increase the RI sensitivity induced by the MT effect at a certain n_{ext} , we need to deposit an overlay with optimized properties, i.e., with appropriate n and thickness [20]. However, it is known that when a high- n overlay is deposited, the resonances at the DTP separate and as a result, the RI sensitivity decreases significantly [8]. In turn, reduction of the diameter of the fiber cladding leads to the opposite phenomenon, i.e., tuning the working point back toward the DTP and increasing the RI sensitivity [7]. In order to benefit from both the MT and DTP effects, the properties of the fiber cladding have first to be optimized by etching [16]. The spectral response of the LPG with slightly etched cladding resulting in a DTP at $n_{ext} \approx 1.429$ RIU and 1.439 RIU is shown in Fig. 1(a) and 1(b), respectively. The etching rate for the SiO_2 glass and applied HF solution has been estimated on reference samples to 30 nm/min [7]. Label-free biosensors require their highest sensitivity at $n_{ext} \approx 1.333$ RIU, and the DTP effect at this n_{ext} can be obtained again after high- n nano-overlay deposition.

Evolution of the response of the LPG shown in Fig. 1(a) after coating with 130 nm of Al_2O_3 is presented in Fig. 2(a). Al_2O_3 is considered as a high- n material ($n \approx 1.61$ RIU at $\lambda = 1560$ nm depending on the thickness of the film [19]) able to tune the optical response of the LPG. As shown in Fig. 2(a), an Al_2O_3 thickness of 130 nm makes both the resonances visible again at the lower n_{ext} , but the working point of the LPG is already away from the DTP. Bringing it back to the DTP at $n_{ext} \approx 1.333$ RIU requires a slight reduction of the overlay thickness, achieved through etching. The etching rate for the Al_2O_3 and applied HF solution has been estimated on reference samples to over 200 nm/min [19]. The response to n_{ext} measured after post-deposition etching is shown in Fig. 2(b). According to the results published earlier [19], for the LPG coated with Al_2O_3 nano-film occurrence of the MT effect in water requires thicker overlays ($d \approx 200$ nm) than those deposited here. This means that successful tuning of the grating properties to achieve both the MT and DTP when the LPG is immersed in water depends on deeper etching of the cladding followed by longer Al_2O_3 deposition [16].

Figure 3(a) and 3(b) show the effect respectively of depositing a thicker overlay ($d = 167$ nm) and post-deposit tuning of the LPG with response before deposition as in Fig. 1(b). In this case we investigated the response in a wider spectral range than previously. Again, despite deeper etching of the fiber cladding, the deposition was long enough to tune the LPG above the DTP at $n_{ext} \approx 1.333$ RIU, and the structure required fine-tuning of the overlay thickness. It must be noted that when this LPG is immersed in higher n_{ext} , i.e., 1.3872 and 1.4004 RIU for as deposited and tuned case, respectively, a higher-order mode (LP_{010})

coupling occurs and its DTP can be traced. The LP_{010} coupling is not as efficient as coupling of the lower-order cladding mode (LP_{09}) and the depth of these resonances is only about -10 dBm. A similar effect has been observed when other high- n materials are used for the overlay [15,21].

Wavelength shifts with respect to n_{ext} for all the cases mentioned above are summarized in Fig. 4. It can be seen that for both overlay thicknesses, the post-deposition etching only slightly increased the sensitivity at n_{ext} values close to that of water. Despite the shift in resonance wavelength induced by overlay etching, up to $n_{ext} \approx 1.38$ RIU the sensitivity is similar to that observed when the LPG works away from the DTP. The highest sensitivity is offered by the higher-order cladding mode (LP_{010}) which experiences its DTP for the thicker overlay ($d = 167$ nm) at $n_{ext} \approx 1.387$ RIU and reaches sensitivity of above $9,300$ nm/RIU. The sensitivity is over 4-fold higher than that measured for the LP_{09} mode at a lower n_{ext} and with a thinner overlay. When the overlay is thinner, the DTP and high sensitivity range are shifted towards higher n_{ext} .

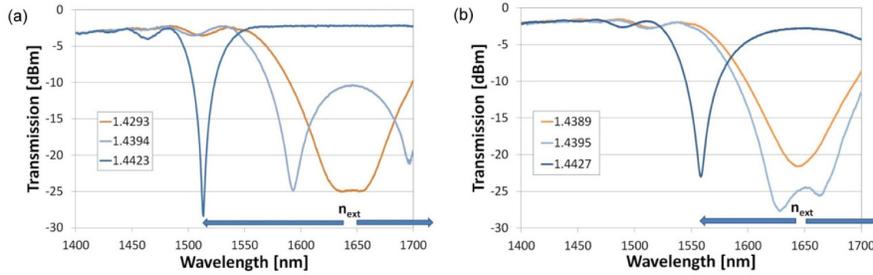


Fig. 1. Effect of etching the fiber cladding on an LPG spectral response to n_{ext} , where (a) shows results after shorter immersion in HF than in the case of (b).

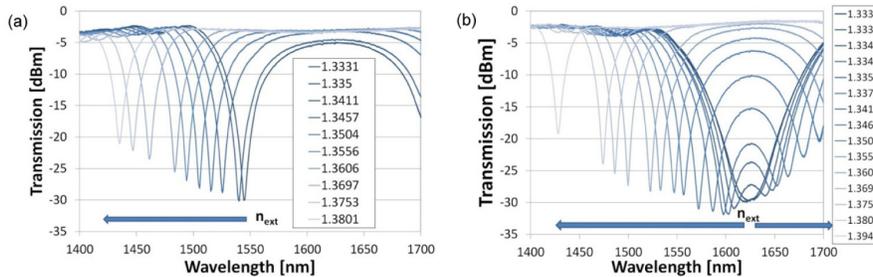


Fig. 2. Response of the etched LPG (coupling of LP_{09} cladding mode) shown in Fig. 1(a) to n_{ext} after (a) deposition of 130 nm thick Al_2O_3 overlay, and (b) after post-deposition HF etching of the overlay down to DTP at $n_{ext} = 1.333$ RIU.

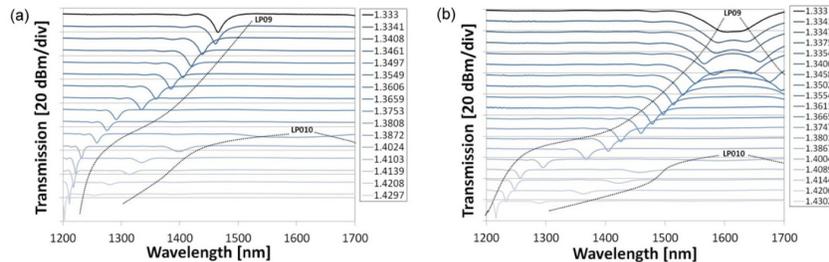


Fig. 3. Response of the etched LPG (coupling of LP_{09} and LP_{010} cladding modes) shown in Fig. 1(b) to n_{ext} where (a) shows results after deposition of 167 nm thick Al_2O_3 overlay, and (b) after post-deposition HF fine-tuning up to DTP at $n_{ext} = 1.333$ RIU.

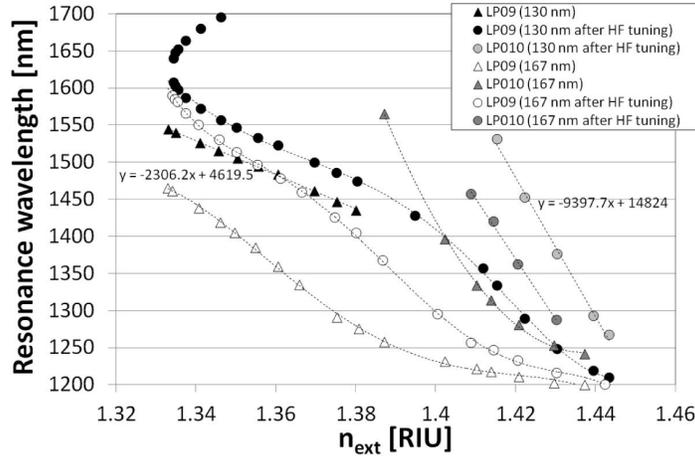


Fig. 4. Resonance wavelength shift vs. n_{ext} for LPG coated with 130 and 167 nm thick Al_2O_3 overlays, as well as after its post-deposition fine-tuning. The effect produced by coupling of the LP_{09} and LP_{010} modes is shown.

Next, we etched the fiber cladding even deeper, down to the point at which the resonances observed in Fig. 1 were not visible any more, even at n_{ext} above 1.45 RIU. Then, the LPG was again coated with a layer of Al_2O_3 , this time 167 nm thick. The results obtained in this case (2nd deposition on deeper etched fiber) are shown in Fig. 5(a), and compared to the effects of the same thickness nano-overlay deposition on the less deeply etched LPG (1st deposition) in Fig. 5(b). It can be seen that deeper etching of the cladding led to reaching the DTP for the LP_{09} and LP_{010} cladding modes at n_{ext} above 1.38 and 1.42 RIU, respectively. At the same time, the sensitivity was further increased for both of the cladding modes. Deeper etching of the fiber cladding also allowed us to trace the shift of the LP_{08} cladding mode appearing in water at $\lambda \approx 1,200$ nm. Due to the work away from the DTP, for this mode the shift is similar to shifts observed for lower-order cladding modes [19].

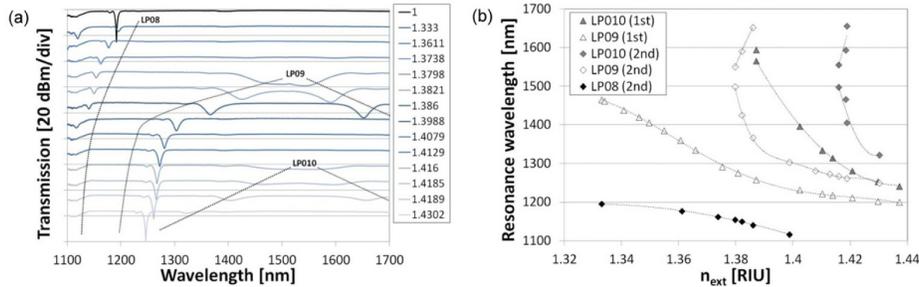


Fig. 5. Response to n_{ext} for LPG etched deeper than for effect shown in Fig. 1(b): (a) spectrum evolution after 2nd deposition of 167 nm thick Al_2O_3 overlay, and (b) resonance wavelength shift after 1st and 2nd 167 nm deposition on shallower and deeper etched fiber cladding, respectively.

Finally, the thickness of the overlay was increased to $d = 182$ and 201 nm. The results obtained for these overlays deposited on a more deeply etched LPG are shown in Fig. 6. It can be seen that the increase in overlay thickness shifts the DTP of the modes towards the lower n_{ext} s, and for Al_2O_3 at $d = 201$ nm, the highest sensitivity is obtained at $n_{ext} \approx 1.34$ RIU. This value correlates well with the results reported earlier in [19], where lower-order cladding modes were traced and the MT was reached at a similar n_{ext} . It must be noted that the etching of the cladding combined with nano-overlay deposition significantly shifts the DTP towards lower wavelengths (Fig. 6(b)).

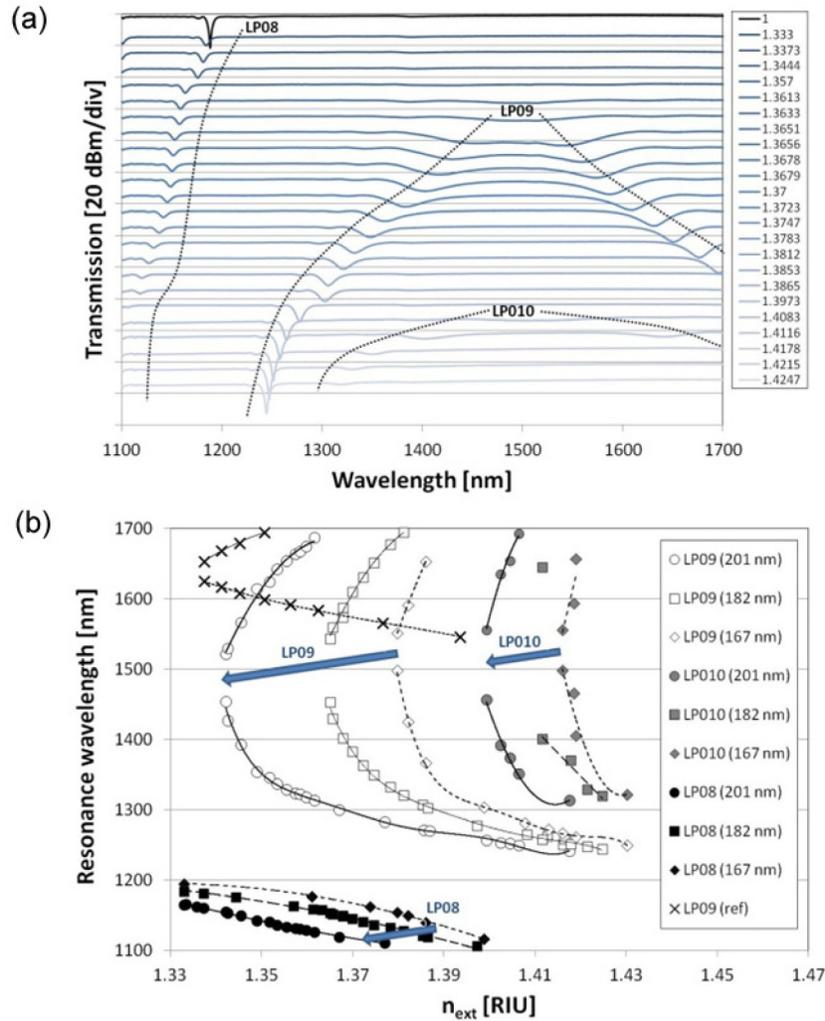


Fig. 6. Response to n_{ext} for deeper etched LPG, where (a) shows the spectrum evolution after deposition of 182 nm thick Al_2O_3 overlay and (b) compares resonance wavelength shifts after 167, 182 and 201 nm depositions on the same LPG. Arrows show evolution of the response with increase of the overlay thickness. The response of a reference LPG before etching and deposition is shown for comparison.

Figure 7 summarizes the results of our RI sensitivity measurements. It can be seen that nano-coated LPGs may offer RI sensitivity reaching over 40 000 nm/RIU. The values were calculated by tracing only one of the pair of resonances, i.e., the one shifting towards lower wavelengths with n_{ext} . These values can even be doubled when the spectral difference between the resonances is taken into account. The high sensitivity range is limited for each of the investigated cases and the sensitivity decreases rapidly with n_{ext} . However, when the traced resonances lose their sensitivity at higher n_{ext} , a new pair of higher-order cladding modes experience the DTP. Appearance of those resonances has been observed for nano-overlays with higher n than that of Al_2O_3 , and with only slight over-etching of the cladding, as previously reported for an overlay of TiO_2 [21].

The results of the present study confirm our previous findings, supported by numerical analysis, that LPG-based high-sensitivity refractometers can only be designed for a narrow range of n_{ext} [16]. In this work, an optimized nano-coated LPG offered sensitivity exceeding

20,000 nm/RIU for n_{ext} close to that of water. This result was obtained for the LP₀₉ cladding mode when the LPG was first etched to obtain a DTP for n_{ext} exceeding 1.45 RIU and was then coated by an Al₂O₃ overlay 201 nm thick.

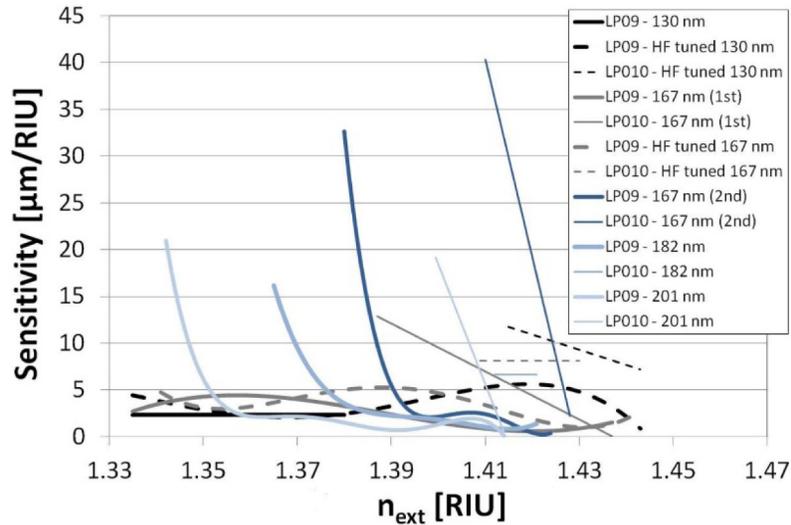


Fig. 7. Summary of the sensitivity of the LPG sample at all stages of the experiment calculated as a first derivative of polynomial fit of the measured results. Here, only resonances shifting towards shorter wavelengths were taken into account.

4. Conclusions

In this work, we report results of experiments on improving the RI sensitivity of LPGs by fiber cladding etching and ALD deposition of a thin Al₂O₃ overlay. At the investigated conditions, the LPG works at the DTP and simultaneously uses the MT effect for the higher-order cladding modes (LP₀₉ and LP₀₁₀). For shallow etching of the cladding followed by the deposition of an overlay of up to 167 nm in thickness, the post-deposition fine-tuning of overlay thickness resulted in a significant increase in RI sensitivity mainly at the DTP of the LP₀₉ cladding mode. In the higher n_{ext} range, an appearance of the DTP corresponding to LP₀₁₀ was noticed, for which the RI sensitivity defined for one resonance exceeded 9,000 nm/RIU. Deeper etching of the cladding followed by deposition of thicker overlays (up to 201 nm thick) made it possible to reach RI sensitivity of over 40,000 nm/RIU in a narrow RI range. Sensitivity exceeding 20,000 nm/RIU was obtained in an RI range suitable for label-free biosensing applications. It must be noted that the sensitivity values can be doubled when the spectral distance between the resonances at the DTP is considered. Moreover, taking into account both the periodical appearance of the DTP for the higher-order modes with increases in RI and the dependence of the DTP on overlay thickness, we believe that an in-line configuration of LPGs etched at the same spot with overlays of different thickness may be applied as a very precise refractometer working in a broad RI range. According to our knowledge, the obtained sensitivities are the highest experimentally achieved to date. Finally, thanks to the high values of sensitivities obtained, the usual units of nm/RIU may be rescaled for simplicity of notation to μm/RIU.

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