



Full length article

# Temperature and refractive index sensing with Al<sub>2</sub>O<sub>3</sub>-nanocoated long-period gratings working at dispersion turning point

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## ABSTRACT

In this work we discuss the effect of nanocoating a long-period grating (LPG) with aluminum oxide (Al<sub>2</sub>O<sub>3</sub>) on both its refractive index (RI) and its temperature (T) sensitivity. Two LPGs, one coated and one uncoated, were optimized to work at the dispersion turning point (DTP) of the higher-order cladding modes, where the sensitivities are the highest. The DTP was reached by two methods – wet etching of the fiber cladding and use of an optimized Al<sub>2</sub>O<sub>3</sub> nanocoating applied by atomic layer deposition (ALD). In both cases we show a significant increase in RI sensitivity at the DTP. Thanks to an additional mode transition (MT) effect, the RI sensitivity of the coated LPG reaches about 8200 nm/RIU in the range  $n_D = 1.333\text{--}1.345$  RIU traced for only one resonance of the pair. This is more than 3.5 times higher than for the non-coated LPG. Moreover, T sensitivity for the Al<sub>2</sub>O<sub>3</sub>-coated LPG was found to be 2.5 times higher than for the non-coated LPG. When the MT effect alone is applied, i.e., the LPG works away from the DTP, the nanocoated LPG may offer higher RI and lower T sensitivity than a standard non-coated LPG working at the DTP. The differences in sensitivity are attributed to the order of coupled cladding modes and the thermo-optic coefficients of both Al<sub>2</sub>O<sub>3</sub> and the external medium. We believe that by proper selection of the nanocoating material, the T sensitivity can be greatly reduced, a major advantage for future biosensing applications of LPGs.

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## 1. Introduction

Optical fiber sensors, due to their numerous advantages including small dimensions, immunity to electromagnetic radiation, and usually relatively simple fabrication processes, have already been widely investigated and applied [1]. The sensors based on optical fiber gratings, i.e., fiber Bragg gratings (FBGs) and long-period gratings (LPGs), are made by periodic modulation of the refractive index within the core of a single-mode optical fiber [2]. In an LPG, the modulation induces coupling between the core mode and the cladding modes, resulting in the appearance of a series of resonance attenuation bands in the grating's transmission spectrum. When the properties of the core, cladding or external medium vary, the resonances experience a spectral shift [3]. Sensitivity to a certain measurand is then typically defined as a spectral shift of the resonance wavelength vs. the measurand [4].

LPGs have already been used for such applications as temperature (T) [5] and pressure [6] sensing, where the difference between the thermo- and pressure-optic coefficients respectively of the

cladding and core materials has a major impact on the sensitivities [6]. Due to coupling of the cladding modes, there is also a significant dependence between the properties of the external medium and the spectral response of the LPG [7]. For this reason, the LPG may be highly sensitive to the external refractive index (RI). Besides the refractometric applications, where high RI sensitivity is reached, the LPG can be used as a universal label-free biosensor targeting biomolecules of different sizes, such as bacteria [8], viruses [9], proteins [10], or toxins [11]. In the label-free sensing approach the sensor is capable of monitoring a biological film growth on the LPG surface, which corresponds to an increase in the RI near the LPG surface [12]. For label-free biosensing applications the LPG must offer high RI sensitivity when working at RI values close to that of water ( $n_D = 1.3330$  RIU).

When optimized RI sensitivity is considered, high-sensitivity conditions are known to occur when the dispersion curve of the cladding mode for a particular effective RI experiences a turning point [4]. At dispersion turning point (DTP) conditions for each of the cladding modes, a characteristic pair of resonances can be observed in the LPG transmission spectrum. The resonances in the vicinity of the DTP shift in opposite directions under certain external influences. In particular, an increase in the external RI is

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followed by an increase in the spectral distance between the two resonances, while for T the direction of the change depends mainly on the properties of the core and the cladding material and can be positive or negative [13]. In addition to the DTP effect when high-RI sensitivity is considered, a mode transition (MT) phenomenon is also known that takes place when the LPG is coated with a high-RI thin coating [14]. Depending on the properties of the coating it can then guide a mode and thus induce transitions of the other cladding modes. At such transition conditions, which depend on the external RI, the RI sensitivity can be greatly enhanced. A high sensitivity in the specified range of RI can be achieved by precise adjustment of the thickness and the optical properties of the coatings [15,16].

Considering both the DTP and the MT effects, the LPG sensitivity to variations in external RI can be improved even more [17,18]. In our previous work [19] we demonstrated how to optimize the RI sensitivity taking into account both these effects by etching the fiber cladding before applying the nanocoating. Both these processes require very high precision and may make the sensor fabrication process relatively complex [20]. However, following results presented in [17], it is known that the DTP and MT effects can also be obtained solely by a nanocoating deposition.

In the present work we investigate and compare in terms of RI and T sensitivities the two methods for tuning LPG working towards DTP, namely etching of the fiber cladding and deposition of a nanocoating. These two types of processing are currently the most often employed for various RI sensing purposes, including label-free biosensing. As a nanocoating, we applied an aluminum oxide ( $\text{Al}_2\text{O}_3$ ) thin film with carefully selected properties. The  $\text{Al}_2\text{O}_3$  was deposited by the atomic layer deposition (ALD) method which gives precise control of the coating properties on the whole length of the grating [20]. The  $\text{Al}_2\text{O}_3$  shows high hardness, as well as high temperature resistance. As discussed above, thanks to nanocoating deposition to induce the MT effect, the RI sensitivity can be greatly enhanced in certain RI ranges. However, the T sensitivity of a nanocoated LPG optimized towards DTP has never been reported or compared to that of an LPG working solely at DTP. The effect of cross-sensitivity to T when the external RI is measured is critical for mature applications of nanocoated LPGs, and that is why we strongly believe that the issue is worth further investigation.

## 2. Experimental details

The LPG sensors in our experiments were manufactured using standard germanium-doped Corning SMF-28 single-mode optical fiber. The process for fabricating the LPGs included: fiber loading with hydrogen, UV irradiation with an amplitude mask (period of 226.8  $\mu\text{m}$ ) and annealing in order to stabilize the optical properties of the LPGs [21]. The first set of LPGs was tuned by hydrofluoric (HF) acid etching up to the DTP for an external RI close to that of water ( $n_D = 1.3330$  RIU) [11]. On the second set of LPGs, an  $\text{Al}_2\text{O}_3$  film was deposited with the ALD method using the Cambridge NanoTech Savannah S100 system [22]. For the deposition process, water and trimethylaluminum (TMA) precursors were used. Thickness of the  $\text{Al}_2\text{O}_3$  film was controlled by the number of ALD cycles, which was set up to 2100. The optimized  $\text{Al}_2\text{O}_3$  film thickness made it possible to achieve both DTP and MT effects.

Spectral response of the LPGs was investigated in the wavelength range 1300–1700 nm using a supercontinuum white light laser source (Leukos SM30) and a spectrum analyzer (Yokogawa AQ6370B). The RI sensitivity was measured by immersing the LPG in a glycerin/water solution with an RI range of  $n_D = 1.333$ –1.345 RIU. The  $n_D$  of the solutions was determined using an automatic refractometer (Reichert AR200). The temperature sensitivity

was measured for LPGs immersed in water by tuning their T in the range from 5 °C to 50 °C using a home-made thermoblock equipped with Peltier modules. The LPGs were also mounted in a special holder to prevent evaporation of water and to maintain the fiber tension during all the experiments.

The sensor's spectral response was analyzed numerically using Optiwave Optigrating software v4.2.2. The LPG model assumed properties of the LPG and coating as reported in [13] and [22], respectively. The RIs of water and  $\text{Al}_2\text{O}_3$  were taken to be 1.318 and 1.611 RIU at  $\lambda = 1550$  nm. No allowance was made for possible change in the optical properties of the coating as a result of change in its thickness within the investigated coating thickness range (200–300 nm) [22].

## 3. Results and discussion

Three LPGs working in different conditions were designed for this experiment: (1) an etched LPG reaching the DTP for an external RI close to that of water, (2) a non-etched grating with nanocoating thickness optimized to obtain both MT and DTP conditions, and (3) an LPG with a slightly thicker coating, where the MT phenomenon still affects the spectral response, but the structure works away from the DTP [20]. The results of numerical analysis of these three designs, assuming water as the external medium, are shown in Fig. 1. When properties of the fiber and the LPG are assumed as in [13], for the LPG (period of 226.8  $\mu\text{m}$ ) in the investigated spectral range just one resonance is observed at  $\lambda = 1230$  nm and this is due to coupling of the  $\text{LP}_{0,9}$  cladding mode. As a result of etching the fiber cladding to reduce its radius by about 5  $\mu\text{m}$ , the resonance shifts towards a longer wavelength and the mode experiences its DTP in the wavelength range above 1600 nm. However, when the LPG is instead given a coating with an RI of 1.611 RIU,  $\text{LP}_{0,9}$  shifts towards a shorter wavelength and a higher cladding mode ( $\text{LP}_{0,10}$ ) appears in the range between 1500 and 1600 nm. The spectral distance between the resonances for  $\text{LP}_{0,10}$  increases significantly with the coating thickness. It is evident that, depending on the approach (etching or deposition), the DTP can be reached, but for a different order of cladding modes. The three LPG variants are further analyzed experimentally.

### 3.1. RI sensitivity

The transmission spectrum of the investigated LPGs before any processing when immersed in water is shown in Fig. 2. The experimental spectrum agrees well with numerical analysis shown in Fig. 1. The effect on spectral response induced by an increase in external RI for the obtained LPGs is compared in Fig. 3. In all cases

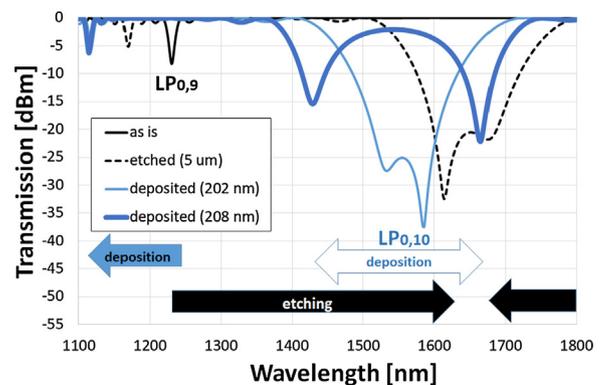


Fig. 1. Numerical analysis of spectral response of the LPG (226.8  $\mu\text{m}$  in period) to etching (5  $\mu\text{m}$  reduction in radius) or nanocoating deposition (increase in coating thickness to 202 and 208 nm). Arrows show progress for each of the processes.

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