Three-hole microstructured optical fiber for efficient Fiber Bragg Grating refractometer

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CEA LIST, Centre d’Etudes de Saclay, 91191 Gif-sur-Yvette Cedex France

Philippe Roy, Jean-Louis Auguste and Dominique Pagnoux

Xlim, UMR CNRS 6172, 123 Avenue Albert Thomas, 87060 Limoges Cedex France

Wilfried Blanc and Bernard Dussardier

LPMC / FOA, UMR-CNRS 6622, Université de Nice Sophia-Antipolis, Parc Valrose, 06108 Nice France

We present a photosensitive three-hole microstructured optical fiber specifically designed to improve the refractive index sensitivity of a standard Fiber Bragg Grating (FBG) sensor photowritten in the suspended Ge-doped silica core. We describe the specific photowriting procedure used to realize gratings in such a fiber. We then determine their spectral sensitivity to the refractive index changes of material filling the holes surrounding the core. The sensitivity is compared to that of standard FBGs photowritten in a six-hole fiber with a larger core diameter. We demonstrate an improvement of the sensitivity by two orders of magnitude and reach a resolution of $3 \times 10^{-5}$ and $6 \times 10^{-6}$ around mean refractive index values of 1.33 and 1.40, respectively.

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Thanks to their attractive propagation characteristics, photonic crystal fibers have gained increased interests in R&D laboratories coming from almost all areas of optics and have found applications in numerous research fields such as non-linear optics [1], chromatic dispersion management [2], high optical power transmission, and sensors [3]. This paper focuses on the development of a FBG-based refractometer using a microstructured fiber whose geometrical properties have been optimized in order to improve the evanescent field overlap with any medium inserted into its holes. We demonstrate a resolution of $3 \times 10^{-5}$ at a refractive index equal to 1.33 simply by using a standard FBG whereas specific procedure (cladding etching or polishing) and/or sophisticated gratings (long period or tilted fiber gratings) have to be implemented to reach similar resolution when using conventional silica fibers (either singlemode or multimode) as sensing platform [4,5].

In a previous study, we had photowritten fiber Bragg gratings in a six-hole Microstructured Optical Fiber (MOF) and determined the wavelength shift of the Bragg resonance versus the refractive index of a liquid inserted into its channels. For a refractive index close to 1.33, the refractive index resolution reached $4 \times 10^{-3}$ r.i.u. (refractive index unit) [6]. In this paper, we present a MOF with a design selected to optimize the interaction (i.e. the overlap) between the evanescent part of the guided electromagnetic field and the medium inserted into the holes. Recently, a similar microstructured pattern, called steering-wheel fiber (SWF), has been numerically investigated: some modelling results suggest a significant overlap between the holes and the electromagnetic field, and hence the interest of such a structure for evanescent-field
based sensing [7]. In our study, a FBG is photowritten in the core of the fiber and we determine its spectral sensitivity to the refractive index of liquid filling the holes. We compare these results to those obtained with the six-hole fiber previously studied [6].

The preform of the MOF is manufactured by means of the usual stack-and-draw technique using silica capillaries and rods of a few millimeters in diameter. For a Ge-doped core MOF, we replace the central silica rod by a Ge-doped silica rod of equal diameter. The Ge-doped rod used for making the fibers core was extracted from a MCVD preform by means of a mechanical machining followed by a HF acid attack. Then a conventional drawing tower was used to draw the microstructured fiber. The resultant fiber is constituted by a single ring of three air holes, surrounding a Ge-doped core of 9 µm² in area (see Erreur ! Source du renvoi introuvable.). The holes area is around 1400 µm². The refractive index contrast of the Ge-doped core with respect to the pure fused silica cladding is equal to 9 x 10⁻³. Such fiber is expected to be multimode in the 1.5 µm spectral range.

The FBG photowriting is performed using a Lloyd mirror interferometer setup including a CW frequency-doubled argon ‘FreD’ laser emitting at 244 nm. When using standard singlemode fiber, the fiber photosensitivity is enhanced prior to the photowriting step by hydrogen-loading the fiber at 180 bar during two weeks at room temperature. With this protocol, we did not succeed in photowriting Bragg gratings in the core of the SWF with a reflectivity higher than 20%. This rather low reflectivity is attributed to the hydrogen desorption during the experiment. This delay, between the moment at which we pull the fiber out of the hydrogen chamber and the moment at which we photowrite the grating, is of only a few minutes. However, it is enough to allow a significant hydrogen desorption. The small core diameter and the large air fraction both contribute to a fast desorption rate of the hydrogen photosensitizing the fiber core: beyond a
delay of fifteen minutes we never succeeded in photowriting any grating. To overcome this difficulty, we followed the procedure proposed by Beugin et al. [8]: two standard singlemode fibers were spliced to each extremity of the SWF in order to realize a kind of gas cell before we put this device in the hydrogenation chamber during two weeks at 180 bar and 25°C. By this way, an overpressure of hydrogen was kept around the core and we succeed in photowriting FBGs with a reflectivity at the Bragg resonance that reached 65 % (the full width at half maximum being around 300 pm). The Bragg resonance as well as other higher order resonances can be easily distinguished on the FBG transmission spectrum (see Figure 2). This means that, as expected, the fiber had a multimode behaviour in the 1.5 µm spectral window.

The refractive index resolution corresponds to the smallest detectable refractive index change that the sensor can detect. To estimate the resolution, we first measured the sensitivity of the sensor at several refractive index values. Then we deduced the resolution from the minimum wavelength shift that we are able to detect: a one picometer wavelength resolution is a reasonable value as explained in [9]. To determine the sensitivity of the FBG with respect to the refractive index, we inserted several calibrated refractive index liquids (perfluorocarbon and/or chlorofluorocarbone oils developed at Cargille laboratories) into the holes of the fiber. The liquids used have a refractive index value covering a range from 1.29 to 1.43 (at 1550 nm and 25°C). These oils were successively inserted by capillarity in the microstructuring channels. We monitored the Bragg resonance spectral evolution versus the liquid refractive index using a narrow bandwidth external cavity tunable laser diode. The results of the measurements are shown on Figure 3 with those from a similar experiment conducted with a six-hole Ge-doped core photosensitive fiber [6].
Assuming a picometric spectral resolution for the Bragg wavelength measurement, we deduced from these curves the refractive index resolution for the two fibers around different refractive index values (see Table 1). With a liquid index close to 1.33, the refractive index resolution reaches $3 \times 10^{-5}$ for the three-hole fiber, whereas we obtained $4 \times 10^{-3}$ for the six-hole fiber. Thus the refractive index resolution is improved by two orders of magnitude. It is improved by a factor of 30 over the range 1.40-1.41. When the refractive index value of the liquid in the holes is beyond 1.41, the fundamental mode is no more guided in the core of the SWF and an optimum sensitivity is reached (asymptotic behaviour). For the six-hole fiber, refractive index measurements can be performed up to 1.45 with a resolution of $6.8 \times 10^{-6}$.

The differences between the two fibers come from the smaller core of the SWF (9 $\mu$m$^2$ in area with respect to 130 $\mu$m$^2$ for the six-hole one). Due to this small area, the electromagnetic field is less confined within the core of the SWF and it extends farther into the holes: hence we observe a greater interaction between any liquid filling the holes and the evanescent part of the field. This explains the increased sensitivity and hence the better resolution obtained with the SWF at low refractive index values, especially around 1.33. Hence such a design is particularly well suited for applications requiring refractive index measurements in aqueous media, as it is the case for biosensors.

In conclusion, a three-hole Ge-doped core photosensitive microstructured fiber (steering-wheel fiber SWF) has been designed and fabricated in order to increase the refractive index resolution of a Fiber Bragg Grating-based refractometer at low refractive index values, typically around those of aqueous media. Hydrogen desorption and the related decrease of the fiber photosensitivity have been overcame by splicing and hence closing the extremity of the microstructured fiber using standard singlemode fibers. FBGs have been photowritten in the core
(9 \ \mu m^2 \text{ in area}) \text{ of the SWF, presenting a reflectivity of 65 \% \text{ at the Bragg wavelength. We have}} \\
\text{shown an improvement of the refractive index resolution of FBGs realized in such a fiber by two orders of magnitude around 1.33 with respect to similar FBGs written in a six-hole fiber with a}} \\
\text{photosensitive core that is 130 \ \mu m^2 \text{ in area. Assuming a picometric resolution for the Bragg wavelength measurement, the minimum detectable refractive index change at 1.33 is equal to } 3 \times 10^{-5}. \text{ Such a fiber is particularly well suited to develop a bio-sensing platform relying on the}} \\
\text{well-established Fiber Bragg Grating-based sensor technology. The following correlative challenging step will consist of bio-functionalizing the inner walls of the channels for the}} \\
\text{selective detection of biomolecules such as proteins. Moreover the overall performances may also be increased through fine tuning of the fiber design and also through the use of core to}} \\
\text{cladding modes coupling using tilted FBGs [10].}

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List of figures and figure captions

Figure 1
Microscope image of the manufactured SWF.

Figure 2
Bragg peak
Coupling to higher order modes
Transmission spectrum of FBG photowritten in the three-hole fiber over a spectral window of 50 nm, showing spectral resonances toward high order modes. The inset shows a zoom on the Bragg wavelength itself.

Figure 3

Wavelength shift of the Bragg resonance *versus* the refractive index of the index liquid for the six-hole (star) and for the three-hole (triangle) photosensitive microstructured fiber.
List of tables and table captions

<table>
<thead>
<tr>
<th>Refractive index</th>
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<td>$\sim 1.33$</td>
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<tr>
<td>$\sim 1.45$</td>
<td>-</td>
<td>$6.8 \times 10^{-6}$</td>
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Table 1

Refractive index resolution obtained for the SWF and the six-hole fiber.