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Refractometric sensing with plasmonic tilted Bragg gratings in different fiber types

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ABSTRACT

Tilted fiber Bragg gratings (TFBGs) coupled to the surface plasmon resonance (SPR) phenomenon represent a powerful sensing solution especially for biodetection purposes. Indeed, the excitation of plasmonic waves at the metal-coated surface of the sensor brings a significant improvement in terms of surface sensitivity. Until now, most of experimental results involving SPR have been obtained by using TFBGs inscribed in standard single-mode optical fibers (SMF), which requires the use of a polarizer to select the right state of polarization for plasmonic excitation. Investigations for the development of even more sensitive or robust sensing tools remain a topic of current interest. In this study, the bulk and surface refractometric sensitivities of plasmonic TFBGs photo-inscribed in multimode fiber (MMF) are compared to the one of standard SMF. Gold-coated TFBG in SMF exhibits a sensitivity value of ~102 nm/RIU. Plasmonic MMF TFBGs are more sensitive with a value of ~124 nm/RIU (enhancement of ~22 %) and open the way to multiplexing thanks to the narrowness of their spectral response. Surface refractometry was also assessed through HER2 bioassays (Human Epidermal Growth Factor Receptor-2), a breast cancer biomarker. For that purpose, aptasensors based on anti-HER2 aptamers were developed and tested by using these two fiber types. Similar surface sensitivities were obtained for both fiber types.

Keywords: Tilted fiber Bragg gratings, plasmon, optical fiber, sensing, refractometry

1. INTRODUCTION

In the field of sensing, plasmonic optical grating sensors have found their place as a powerful tool for real-time monitoring of refractive index changes with various applications1–3, especially for bio-chemical detection purposes4–12. This well-established technology in the scientific literature brings a highly sensitive and versatile platform for biosensing since it allows the implementation of a large range of detection strategies. Indeed, the immobilization of various bioreceptors (antibodies, aptamers, etc.) can be achieved to target and detect miscellaneous analytes of interest (DNA, proteins, cells, enzymes, etc.) in a very rapid and efficient way. Optical fiber gratings offer many advantages and lead to the development of micro-scaled, flexible and cost-effective sensors for a label-free, fast and accurate detection13–15. Tilted fiber Bragg gratings (TFBGs) are usually used as refractometers due to their own intrinsic characteristics. A TFBG is defined as a permanent and periodic variation of the refractive index located within the fiber core and featuring a slight angle (1-10°) with respect to the perpendicular to the fiber axis. This structure is generally obtained thanks to a laser photo-inscription coupled to the use of a tilted phase-mask16. This optical and reflective component allows to couple a part of the guided core light into the cladding, the latter interacts then with the surrounding medium making the sensor sensitive to surrounding refractive index (RI) changes. The significant enhancement of the sensitivity is achieved with a surface plasmon resonance (SPR) excitation, made possible after the deposition of a thin and finely tuned metal layer at the grating surface13,17. More specifically, this oscillation of electrons absorbs a part of the cladding light and increases the intensity of the electric field very closely to the fiber surface which is responsible of the high improvement in the refractometric sensitivity. The determination of the RI of solutions is largely used in industries (agri-food industry, chemical processes, etc.) for quality control purposes since its value can be directly related to the sample density, purity or chemical concentration6,18–20. Nevertheless, one of the major applications of plasmonic sensors is when bioreceptors are grafted on to the sensing area, turning it into a biochemical and/or medical sensor. In this context, attempt to push the limit of performances for early detection of pathologies is always a current concern. Most of experimental results generated to date are obtained from the use of standard telecommunication grade single mode fibers (SMF). Higher
sensitivity and/or more robust sensors are of interest for a possible use of this highly-sensitive technology for point-of-care diagnosis. To this aim, we investigate in this work the bulk and surface refractometric sensitivities of plasmonic TFBG sensors inscribed in multimode fiber (MMF) in comparison to SMF. Experiments in bulk refractometry were also conducted with so-called single polarization fibers (SPF) that have a bow tie geometry with a very strong birefringence.

2. MATERIAL AND METHODS

Tilted fiber Bragg gratings (TFBGs) were manufactured within the Ge-doped optical fiber core after a photosensitivity amplification through a hydrogenation process (200 bar, 60 °C, 30 h). The photo-inscription was performed in different optical fiber types: standard telecommunication single mode fiber (SMF-28, Corning), multimode fiber (MMF 50/125, Corning) and bow-tie single polarization fiber (SPF HB1550Z, Fibercore). For that purpose, the NORIA system (NorthLab Photonics, Sweden) featuring an excimer laser at 193 nm and a uniform tilted phase-mask (8° with respect to the perpendicular to the fiber axis) were used. For a long-term stabilization, gratings were thermally annealed at 100°C for 24 h. Prior experiments involving an SPR excitation, the grating area was covered with a thin gold layer (~35 nm) by using a sputtering process (Leica EM SCD 500). To ensure the gold adhesion on the silica fiber surface, the sensitive area was then locally heated at 200 °C for 2 h. Then, refractometric sensitivity was assessed on different sensor configurations (bare and gold-coated) by using Lithium Chloride (anhydrous, 99%, Alfa Aesar) solutions (in ultra-pure water). For bioassays, sensors were biofunctionalized by immobilizing thiolated anti-HER2 ssDNA aptamers (5′-TCT AAA AGG ATT CTT CCC AAG GGG ATC CAA TTC AAA CAG C-S-S-3′) onto the gold-coated surface. First, an aptamer selection by the Systematic Evolution of Ligands by EXponential enrichment (SELEX) method and a synthetization using a MerMade 6 automated DNA synthesizer (BioAutomation USA) were achieved. Then, aptamers were resuspended in TE buffer (Tris-ethylenediaminetetraacetic) 100 µM (Base Pair Biotechnologies) and diluted 1:1 v/v with TCEP (Tris (2-carboxyethyl) phosphine) solution. After a reduction step at 90 °C for 5 min, aptamers were immobilized onto the gold surface for 1h at a concentration of 10.24 µM. Phosphate Buffer Saline (PBS) pH 7.2 from Thermo Scientific was used to reach this concentration. Before testing, the biosensor surface was blocked using 6-mercaptop-1-hexanol (Sigma Aldrich) 5 mM for 30 min in PBS at room temperature. Finally, the developed aptasensors were tested in an increasing range of HER2 concentrations from 10^{-12} to 10^{-6} g/mL. Measurements were performed using a LUNA optical vector analyzer (OVA).

3. RESULTS AND DISCUSSION

3.1 Bulk refractometry using standard single mode fiber

First, the refractometric sensitivity of TFBGs inscribed in SMF was assessed by immersing the sensor into different RI solutions around the 1.343 RI value. Tests were performed on both bare and gold-coated configurations (Figs 1 and 2). In case of a bare TFBG, the sensitivity value was determined by tracking the wavelength shift (in nm) of the cut-off mode as a function of the refractive index unit (RIU) shift in a 15.10^{-4} RIU range with 3.10^{-4} RIU steps. In a given RI range, this mode possesses the closest effective refractive index to the surrounding RI value and is the most sensitive one. For a gold-coated TFBG-based sensor, the presence of an SPR excitation results in an attenuation in the comb-like transmitted amplitude spectrum of the sensor. The demodulation technique of such a refractometer is then based on another approach. In fact, it is impossible to directly track a mode located in the SPR signature wavelength range due to the strong and noisy attenuation of signal amplitude. The detection is then based on the most sensitive mode near this plasmonic feature. The most sensitive mode is typically located between the SPR attenuation and the cut-off mode, the latter has the largest peak-to-peak amplitude to the left of the plasmonic signature. For the gold-coated configuration, sensitivity measurements were achieved in a 9.10^{-4} RIU range with 3.10^{-4} RIU steps (Fig. 2). Bare SMF TFBG (Bare-SMF) exhibits a refractometric sensitivity of 33.73 nm/RIU (R² = 0.999) (Fig. 1(c)) while the gold-coated one (Gold-SMF) shows a value of 102.03 nm/RIU (R² = 0.999) (Fig. 2(c)). A significant improvement of ~200 % is therefore observed. Thanks to the SPR phenomenon, the electric field near the TFBG surface is reinforced, which explains the sensitivity enhancement.
Figure 1. Spectral response of a bare TFBG-based single mode fiber sensor (Bare-SMF) (a) with a zoom around the cut-off mode (b). Wavelength shift (in nm) of the cutoff mode as a function of the RI shift for a bare TFBG-based SMF sensor (c).

Figure 2. Spectral response of a gold-coated TFBG-based single mode fiber sensor (Gold-SMF) (a) with a zoom around the most sensitive mode (b). Wavelength shift (in nm) of the most sensitive mode as a function of the RI shift for a gold-coated TFBG-based SMF sensor (c).

3.2 Bulk refractometry using multimode fiber

Next experiments show the feasibility of grating photo-inscription and SPR excitation in a multimode fiber. Under the same conditions, refractometric sensitivities of bare (Bare-MMF) and gold-coated TFBG-based MMF sensors (Gold-MMF) were assessed. Since sensors using SPR excitation are widely used in the biochemical sensing field, a gold layer
is usually used as a metal layer for stainless and biocompatibility reasons. When results of Figs. 3 and 4 are compared to the ones of previous figures, we can observe that MMF-based sensors are characterized by narrower spectra (30-40 nm) than a spectral response recorded for an SMF-based sensor (90-100 nm).

Figure 3. Spectral response of a bare TFBG-based multimode fiber sensor (Bare-MMF) (a) with a zoom around the cut-off mode (b). Wavelength shift (in nm) of the cutoff mode as a function of the RI shift for a bare TFBG-based MMF sensor (c).

Figure 4. Spectral response of a gold-coated TFBG-based multimode fiber sensor (Gold-SMF) (a) with a zoom around the most sensitive mode (b). Wavelength shift (in nm) of the most sensitive mode as a function of the RI shift for a gold-coated TFBG-based MMF sensor (c).

In addition, the cladding mode coupling is not effective in case of an MMF configuration for a wavelength range located between ~1560 nm and ~1588 nm (Bragg wavelength) due to the larger value of the fiber core. Indeed, the used MMF
features a 50 µm core diameter against 8.2 µm for the SMF. For both fiber types, the total core-cladding diameter is 125 µm without the polymer jacket. As a result of this, it appears that the use of MMF for grating inscription will be suitable for multiplexing purposes in aqueous solutions. Two TFBGs can be easily and separately interrogated at the same time in the C+L wavelength bands, an experiment impossible to conduct with a single mode fiber. Refractometric measurements led to sensitivity values of 56.30 nm/RIU (R² = 0.993) and 124.98 nm/RIU (R² = 0.998) for bare (Bare-MMF) (Fig. 3(c)) and gold-coated TFBGs inscribed in MMF (Gold-MMF) (Fig. 4(c)). In this case, the observed improvement is ~122 %.

### 3.3 Bulk refractometry using bow-tie single polarization fiber

The used single polarization fiber is a bow tie fiber especially designed to strongly attenuate one of the two orthogonal modes and then maintain the propagation of only one state of polarization. Figure 5 shows the spectrum of a gold-coated TFBG inscribed in this fiber (Gold-SPF) and immersed in different solutions. By tracking the most sensitive mode, we observed that a plasmonic SPF TFBG exhibits a refractometric sensitivity of 93.99 nm/RIU (R² = 0.993) (Fig. 5c). For a direct comparison with the other configurations, refractometric sensitivities of the Bragg grating sensors based on the different tested fiber types are gathered in Table 1.

Figure 5. Spectral response of a gold-coated TFBG-based single polarization fiber sensor (Gold-SPF) (a) with a zoom around the most sensitive mode (b). Wavelength shift (in nm) of the most sensitive mode as a function of the RI shift for a gold-coated TFBG-based SPF sensor (c).

Table 1. Comparison of refractometric sensitivities in nm/RIU for the different tested plasmonic TFBG sensors.

<table>
<thead>
<tr>
<th>Fiber type</th>
<th>Sensitivity [nm/RIU]</th>
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</thead>
<tbody>
<tr>
<td>Bare-SMF</td>
<td>33.73</td>
</tr>
<tr>
<td>Bare-MMF</td>
<td>56.30</td>
</tr>
<tr>
<td>Gold-SMF</td>
<td>102.03</td>
</tr>
<tr>
<td>Gold-MMF</td>
<td>124.98</td>
</tr>
<tr>
<td>Gold-SPF</td>
<td>93.99</td>
</tr>
</tbody>
</table>

The sensitivity value of a plasmonic TFBG-based SPF sensor is very close to the refractometric sensitivity of a gold-coated SMF (102.03 nm/RIU), but MMF remains the most sensitive in bulk refractometry (124.98 nm/RIU). Nevertheless, the use of SPF allows to facilitate the propagation of the right polarization state for SPR excitation and then the clean SPR signature is more tolerant to polarization instabilities linked for instance to the movement of optical
fiber cables. This is an interesting added-value in practice, despite the much higher cost for that fiber compared to telecommunication-grade ones.

3.4 Surface refractometry through HER2 bioassays

To compare performances of SMF and MMF-based sensors in term of surface sensitivity, bioassays using biofunctionalized gold-coated fibers were performed on HER2 (Fig. 6). Several biomarker concentrations were tested ranging from $10^{-12}$ to $10^{-6}$ g/mL with a reference measurement in PBS. Apatasensors were immersed in HER2 solutions for 10 min during which the wavelength shift of the most sensitive mode was tracked in order to assess the sensitivity. This shift reflects therefore the protein binding at the sensor surface.

![Figure 6. Spectral response (in wavelength shift) of SMF (grey) and MMF (blue) apasensor for different HER2 concentrations with respect to a reference measurement taken in PBS (black).](image)

As expected, we observe a gradual increase of the apasensor response when the HER2 concentration grows for both sensor configurations. In contrast with bulk refractometric experiments, MMF-based apasensors show a quite similar response to the SMF use in terms of surface detection. To validate the comparison, the computation of the fill factor (i.e. the power fraction) for the considered configurations was done by using the FimmWave simulation software (Table 2). As model, a 1 nm thick sheath around the fiber with a RI value $4 \times 10^{-5}$ higher than the surrounding medium (PBS) was chosen to represent the aptamer layer. A higher power fraction is obtained for the MMF (58.4%) than in the case of the SMF (50.2%) in the surrounding medium while values in the modelled aptamer layer are similar, which explain results obtained in bulk and surface refractometries.

<table>
<thead>
<tr>
<th>Medium</th>
<th>Fill Factor (Percentage)</th>
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<tbody>
<tr>
<td>PBS</td>
<td>50.2%</td>
</tr>
<tr>
<td>Aptamer</td>
<td>0.4%</td>
</tr>
</tbody>
</table>

Table 2. Comparison of the power fraction depending on the medium for the SPR mode.

4. CONCLUSIONS

This study was intended to investigate the performances in terms of bulk and/or surface refractometric sensitivities of plasmonic tilted fiber Bragg grating sensors inscribed in multimode and single polarization fibers. The aim was to compare their use to the typical approach of TFBG sensors, i.e. usually based on grating inscription in standard telecommunication single mode fibers. The advantage of a grating inscription in MMF relies on a better bulk refractometric sensitivity value of ~124 nm/RIU for plasmonic sensing in comparison with the value of ~102 nm/RIU, obtained in case of SMF (enhancement of ~22 %). To study the performances in terms of surface refractometry, biodetection of the HER2 breast cancer biomarker in different concentrations was performed. To this aim, the two gold-coated fiber types were biofunctionalized using anti-HER2 aptamers. It turns out that both configurations lead to a similar behavior for surface detection. The aforementioned results are supported by a fill factor value computation showing the same trend for both bulk and surface sensing approaches. Moreover, the spectral narrowness of the sensor response can make possible the multiplexing of at least two gratings while it is not possible in case of the use of an SMF,
by considering the same wavelength span corresponding to most FBG commercial interrogators (C+L bands). Multiplexing brings the advantage of enabling several measurements at a time which can therefore provide more reliable results.

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