Abstract — This work shows results from an experimental study of the thermal sensitivity of an etched fiber Bragg grating immersed in several different surrounding media. The used FBG has a diameter of \(9.0 \pm 0.5\) \(\mu\)m resultant from the etching process. The device thermal sensitivity shows a decrease when the surroundings refractive index rises, changing from negative to positive values depending on the external medium refractive index and temperature range. Besides that, a noticeable non-linearity is observed on the grating thermal response when the surroundings refractive index rises towards the fiber-cladding index. These behaviors are attributed to the combined effects of the device response to temperature and surroundings refractive index.

Index Terms — etched fiber Bragg grating, refractive index sensitivity, thermal sensitivity, sensors.

I. INTRODUCTION

The use of optical fiber gratings in the sensing field to measure several parameters like temperature, strain and surrounding refractive index was extensively investigated in the past years. The intrinsic characteristics of the fiber, such as reduced size and weigh, electromagnetic immunity and electrical passivity, allied with fiber grating own characteristics like multiplexing capability, wavelength encoding and high sensitivity make the grating sensors especially versatile. Within this context, the oil and gas sector [1] is a particular field of interest for the application of these sensors. A prospective family of optical sensors for chemical and biochemical applications is based on long period gratings (LPG) due to its inherent sensitivity to the refractive index of the surrounding medium [2-3]. However, the relative wide bandwidth of the transmission spectrum limits the performance of the detection techniques and reduces the effectiveness of the multiplexing capability. A further complication arises from nonlinearities in the LPG behavior, as noticed for example in the grating thermal response when the device is immersed in samples with high refractive indexes (e.g. hydrocarbon derived from petroleum) [9], what in turn can result in errors in the measurand obtained from extrapolation.

On the other hand, a noticeable increase in fiber Bragg gratings (FBG) based refractometer investigations has been observed recently. Thinned and micro-structured fiber Bragg gratings have been proposed as chemical sensors [4-5]; D-shaped, single and multimode fiber gratings have also been reported as chemical concentration sensors [6]. However, the FBG present an inherent cross-sensitivity problem due to the dependence of the waveguide’s modal effective refractive index both to
the temperature and to the surrounding refractive index, and many techniques have been implemented to overcome that drawback. Devices using two FBGs being one of them partially etched to modify its characteristics [7], or a non-uniform thinned grating [8], have also been proposed.

In this work the influence of the refractive index sensitivity on the thermal sensitivity of an etched cladding FBG is analyzed for the grating immersed in several surroundings media with different refractive indexes.

II. EXPERIMENTAL

The FBGs are written in a photosensitive optical (FiberCore PS1250/1500) fiber with 125 μm of cladding diameter using a phase mask interferometer illuminated with UV light (244 nm Ar+ laser). The gratings have an estimated length of the 2 mm, derived from the laser spot diameter. The FBGs are chemically etched using an aqueous solution of hydrofluoric acid (HF). The diameter of the FBG sensor is reduced with the immersion of the fiber into the acid solution for about 257 minutes, and after this time interval the etching treatment is neutralized immersing the fiber in a NaOH solution. In order to determine the etching rate a first FBG was completely etched and the evolution of the Bragg wavelength was monitored along the treatment.

The optoelectronic set-up employed in the etched cladding FBG production and characterization uses a conventional Optical Spectrum Analyzer (OSA, 0.1 nm of resolution, ± 5 pm of wavelength stability) and an erbium-doped fiber amplifier as a broadband source to record the grating spectra.

The etched FBG refractive index sensitivity (RIS) is obtained by recording the device spectrum with the grating completely immersed into the different samples: water (n = 1.323), Ethanol (n = 1.354) and several commercial hydrocarbon mixtures (Benzyne, Thinner, half-half mix of Thinner and Turpentine, Turpentine, Kerosene), hereby called HC 1 (n = 1.366), HC 2 (n = 1.387), HC 3 (n = 1.408), HC 4 (n = 1.439) and HC 5 (n = 1.444). To assure no effects of residual liquid contamination, before each measurement with a specific sample, the spectrum of the grating immersed in water was recorded. After the measurement with each sample, to remove any hydrocarbon trace from the grating surface, a 1.5-hour immersion cleaning cycle using Thinner, Ethanol and water was realized, and the grating spectrum in water was compared with the spectrum measured in water before the immersion in each hydrocarbon sample. The refractive index of each sample is measured with a commercial Abbe refractometer with a resolution of 10^{-3} refractive index units (RIU). The measurements are done at three different wavelengths: 589.3 nm from the sodium D-line, 543.5 nm and 632.8 nm from He-Ne lasers, and a three term Sellmeier equation is used to estimate the refractive index at 1550 nm. In the experiments the temperature was monitored and kept constant within (21 ± 0.1) °C.

The thermal sensitivities of the etched FBG are obtained with the device immersed in a container with about 60 ml of each sample. To minimize the influence of the sample evaporation rate on the sensor response the grating was kept about 3 cm below the sample’s surface. In the experiments the device reflection spectrum is recorded after temperature changes of 1 °C in the range between
(23 ± 0.1) °C and (42 ± 0.1) °C.

III. RESULTS AND DISCUSSIONS

The results obtained, monitoring the evolution of the Bragg wavelength during the entire etching treatment, allows finding an etching rate of about 0.45 μm/min (figure 1). It is observed that after a time interval of 279 minutes the reflected spectrum disappears indicating the end of the etching process. In figure 1 it is also presented the evolution of the fiber diameter along the etching treatment.

Fig. 1. Evolution of the Bragg wavelength during the entire etching treatment and the estimated fiber diameter. The line connecting the points is solely a visual aid.

Figure 2 shows the refractive index sensitivity (RIS) of an etched FBG with final diameter of (9.0 ± 0.5) μm. The RIS (dashed line) is obtained by the numerical derivative of an empirical rational equation, (solid line), a, b and c are fitting parameters adjusted to the experimental points.

Fig. 2. Bragg wavelength and refractive index sensitivity versus refractive index of surrounding medium for the etched grating. The marks depict the experimental points, the curves shows the empirical best fit for the Bragg wavelength (solid line) and the calculated RIS (dashed line).
The temperature response of the Bragg wavelength when the device is immersed in each sample is shown in the figure 3 together with a second order polynomial fit of the experimental points. As the refractive index shifts towards the high RIS range, a nonlinear behavior of the wavelength shift versus temperature becomes observable. For samples HC 4 and HC 5 this behavior is markedly nonlinear, whereas the samples water, ethanol, HC 1 and HC 2 behaves linearly, within the experimental uncertainties. The sample HC 3 lies just in the transition between the two behaviors.

![Figure 3. Temperature response of the etched FBG immersed in samples with different refractive indexes. The marks show the experimental points, and the lines depict the respective best fit.](image)

Figure 4 shows the influence of surroundings on the grating thermal sensitivity obtained by differentiating the polynomial fitting function (figure 3) with respect to temperature. The increase of external medium refractive index results in lower average values of thermal sensitivity, within the analyzed temperature range. Nevertheless, the thermal sensitivities become strongly dependent on the temperature for samples with refractive indexes closer to the cladding index. For sample HC5, which presents the most noticeable non-linear behavior, the thermal sensitivity moves from negative to positive values.

Table 1 summarizes the data related to the used samples. Thermal characterization was not carried out for the grating in the air.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Refractive index, n</th>
<th>RIS (nm/RIU), fig.2</th>
<th>Non-linear coefficient, fig. 3 (10⁻¹ pm/°C²)</th>
<th>Angular coefficient (pm/°C²), fig. 4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Air</td>
<td>1.000</td>
<td>0.16 ± 0.01</td>
<td>---</td>
<td>---</td>
</tr>
<tr>
<td>Water</td>
<td>1.323</td>
<td>1.47 ± 0.01</td>
<td>-0.03 ± 0.38</td>
<td>-0.006 ± 0.01</td>
</tr>
<tr>
<td>Ethanol</td>
<td>1.354</td>
<td>2.22 ± 0.01</td>
<td>-0.09 ± 0.38</td>
<td>-0.019 ± 0.01</td>
</tr>
<tr>
<td>HC 1</td>
<td>1.366</td>
<td>2.67 ± 0.01</td>
<td>0.19 ± 0.38</td>
<td>0.039 ± 0.01</td>
</tr>
<tr>
<td>HC 2</td>
<td>1.387</td>
<td>3.88 ± 0.01</td>
<td>0.22 ± 0.38</td>
<td>0.044 ± 0.01</td>
</tr>
<tr>
<td>HC 3</td>
<td>1.408</td>
<td>6.11 ± 0.01</td>
<td>0.46 ± 0.38</td>
<td>0.092 ± 0.01</td>
</tr>
<tr>
<td>HC 4</td>
<td>1.439</td>
<td>16.10 ± 0.01</td>
<td>1.01 ± 0.38</td>
<td>0.201 ± 0.01</td>
</tr>
<tr>
<td>HC 5</td>
<td>1.444</td>
<td>18.77 ± 0.01</td>
<td>3.61 ± 0.38</td>
<td>0.722 ± 0.01</td>
</tr>
</tbody>
</table>
IV. CONCLUSION

In this work is verified experimentally the influence of the surrounding media with different refractive indexes in the thermal sensitivity of an etched FBG with diameter of (9.0 ± 0.5) μm. Within the samples used in the experiments, is observed a decrease in the average thermal sensitivity when the refractive index of surroundings rises. Besides, the results show nonlinearities in the grating thermal response that are more significant when the external medium refractive index is close to the cladding index. For refractive indices above 1.408 (HC3 sample) this nonlinearities must be considered for the sensor calibration, leading to a correct response of the transducer device. These observed behaviors are consequence from the combined influence of three factors of the grating response: the silica and surrounding medium thermo-optic coefficient and the device refractive index sensitivity. For a particular sample, the temperature increase causes a decrease in its refractive index due to the thermo-optic effect and the Bragg wavelength presents a shift to lower wavelengths (see fig. 2). This blue shift is opposite to the red shift resultant from the silica thermo-optic effect. The blue wavelength shift is greater for samples with higher refractive indexes (HC 4 and HC 5) as these samples are in the high RIS range and, as a consequence, present lower average values of thermal sensitivity. In the other hand, for a particular sample the decrease in its refractive index when the temperature rises also causes a decrease in the grating RIS (see in figure 2) and the blue shift causes lower changes in the wavelengths. As a result, the blue wavelength shift contribution is smaller in the high temperature range and the thermal sensitivity increases. This effect is more noticeable for the sample HC 5, that shows a thermal sensitivity negative (−6.4 ± 0.3 pm/ºC) at 23 ºC and positive (8.3 ± 0.3 pm/ºC) at
42 °C, and is an important aspect to avoid misinterpretations resulting from extrapolation of the values outside the measured range. Firstly, the thermal response of sensor must be determined for the medium that the sensor is intended to work with. Secondly, the thermal response within the whole sensor operation temperature range must be known to consider correctly any nonlinear behavior. Finally, if the sensor will be subjected to different media along its operation, the two above described features need to be analyzed carefully for each different sample.

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