Long Period Grating Application for Fuel Quality Control

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Abstract

We determine the volumetric concentration of ethanol mixed in commercial gasoline by using a long period grating written in standard telecommunication fibre optic. The grating is produced by a point to point electric arc discharge of a splicing machine. The spatial period and length of the used grating are, respectively, 649 μ m and 34 mm. The average shift of the wavelength of the resonance peak is about 4 nm when ethanol proportion changes from 24 to 49 %. Such determination is adequate to monitor the mixture of ethanol in gasoline for consumer's quality control applications.

1. INTRODUCTION

Refractive gratings in fibre optic present a large variety of applications both in optical communications and as reliable fibre optic sensors. Optical communications devices like, e.g., wavelength filters, gain equalizers or dispersion compensation modules can be assembled from Bragg gratings. For sensors applications the gratings are mainly used to measure physical parameters like temperature, strain or refractive index¹. Apart from their applications in the measurement, those sensors also have all the intrinsic characteristics of the fibre optics, like passive electrical operation, immunity to electromagnetic interference, high sensitivity, and spectral multiplexing¹. They also show high fusion temperature, low attenuation, reduced weight and small volume. All those characteristics are important for sensing applications. Particularly for applications in Oil and Gas Industry, fibre optic sensors can be remotely operated over a fibre link that can be several kilometres long. This avoid bringing electrical signal to the sensors, reducing potential hazards derived from electrically induced fire or explosion of the combustible products.

Research in the particular class of long period gratings (LPG) attracted much attention in the past years². In comparison with standard Bragg gratings, that have spatial period in the order of a micrometer, the LPG presents a longer spatial period for the refractive index modulation, in the order of hundreds of micrometers. The LPG operates by coupling the fundamental mode in the core of the fibre to co-propagating cladding modes. Standards Bragg gratings couple the fundamental mode with a counter propagating mode in the core, and the large change in the wave vector implies short periods for the grating. For a LPG, the small difference in the co-propagating wave length of a particular coupling to a cladding mode is given by²:

$$\lambda_{\rm m} = \left(n_{co} - n_{cl}^{\rm m} \right) \Lambda \tag{1}$$

where λ_m is the peak wavelength of the resonance band between the core mode and the cladding mode, n_{co} and n_{cl}^m are, respectively, the effective refractive index of the core mode and of that of the *m*-th order cladding mode and Λ is the grating pitch. Since the interaction of the guided mode in the fibre optic occurs with a cladding mode, that is strongly affected by fibre imperfections, micro and macro bending and by the boundary condition at the cladding - external medium interface, light coupled from the core mode leaks out the fibre, leaving several transmission dips in the transmission spectrum, each one corresponding to a specific coupling governed by Eq. 1.

LPG are very useful as sensor when the refractive index of the external medium changes. If the parameter to be measured affects the refractive index, this will also change the matching condition expressed by Eq. 1 and will lead to a wavelength shift of the resonance dip in the LPG transmission spectrum. That occurs because the effective indexes of cladding modes are dependent on the refractive index of the core, cladding and external

medium. For comparison, in the case of Bragg gratings the effective index of the mode in the fibre's core depends on the core and cladding refractive index, so that a change on the external medium index only is perceived when the cladding is almost entirely removed, to expose the evanescent field of the core mode to the external index. As LPG have fast response to the change in the external medium²⁻⁴, their use as sensors of refractive index changes is far more interesting than sensors based on Bragg gratings. Another advantage is that the cladding does not need to be removed, a fact that drastically affects the mechanical properties of the fibre optic⁴.

In this work we show the use of a LPG as a sensor for measuring the refractive index of a mixture of ethanol and gasoline. In Brazil this mixture has legal validity for an ethanol (distilled from sugar cane) proportion up to 24 %. However, as the ethanol has lower price (about 60 %) than gasoline, a common malpractice is to increase ethanol concentration in the mixture that is sold to car owners. Procedures to verify the contents of ethanol in the mixture are required to assure the legal limit and to protect consumer's rights. Although we describe that particular sensor application, its use can be extended to any other kind of contaminant in the gasoline mixture (including other hydrocarbon based products that enter in the final composition of the commercial gasoline).

2. EXPERIMENTAL

The LPG are produced using a technique similar to that described by⁵. A bare fibre with its protective coating removed is inserted between the electrodes of a commercial splicing machine. A small weight is suspended in one of the fibre's extremities to keep a constant longitudinal tension. The other extremity of the fibre is mounted on a computer controlled translation stage. An electrical arc is applied with the splicing machine, using adequate current and time duration settings. After the discharge, the fibre is moved by the required period of the grating, before another arc is applied. After a suitable number of point to point discharges, a periodic pattern is engraved in the fibre's refractive index profile, due to heating activated processes. An optical set-up is used during the writing process to monitor the transmission spectrum through the fibre. When the measured spectrum shows characteristics adequate to the intended application, the process is interrupted. Usually 40-70 points were necessary to form a grating for the described sensor. The advantage of using the electrical arc is that no special fibre (hydrogenated of pre sensitised) is required. Also, splicing machines are a very common apparatus in optical fibre laboratories.

Measurements for the characterization of the LPG and to determine its sensitivity to external index changes are made using a halogen lamp, whose light is focused on the entrance slit of a monochromator. After the exit slit, light is modulated by a mechanical chopper and launched into the fibre using a 40X microscope objective. The light on the fibre's output is collected by another 40X objective onto an InGaAs photo detector. The electrical signal from the photo detector is fed to a lock-in amplifier, synchronized to the chopper's modulating frequency. A personal computer controls the fibre positioning system⁶, the monochromator grating position and collects the resulting data through an Analogue to Digital Converter card. The gratings used in this work were written using a current of 12 mA in the electrical arc of the splicing machine, with a time duration of 0.5 s. The resolution of the translation stage is 5 μ m.

The response of the sensor to the external medium index is measured as follows. First the fluid in laid over the fibre (covering all the region where the LPG resides) and a cover glass is used to insure that the LPG remains inside the liquid during all the measurements. Then transmission spectra are recorded as a function of the liquid mixture, using the same equipment as described in the previous paragraph. Initially a few calibration measurements were taken using mixtures of several fluids whose refractive index were known. A grating with 40 periods (pitch of 649 μ m) is used and the corresponding results are shown in Fig. 1. It can be seen that the larger change in the resonant wavelength occurs when the refractive index of the external medium is close to the cladding refractive index³⁻⁴. A spectral shift of 7 nm in the refractive index range from 1.333 and 1.426 is obtained, which translates as an average resolution⁷ of 0.0116 nm⁻¹.

Apart from the wavelength shift when the refractive index of the external medium changes, LPG also present a reduction in the peak attenuation of the resonance band³. This can also be seen in our measurements, as shown in Fig. 2, where there is a comparison between the spectra taken with pure water and the spectra taken with a mixture of water and glycerine covering the fibre. The later mixture has a refractive index that is closer to the cladding index, causing greater mismatch between the core and cladding modes. This fact reduces the power coupled from core to cladding modes. The spectra of Fig. 2 were taken with the same grating used to obtain the data of Fig. 1.



Fig. 1. Peak position of the principal attenuation dip in the transmission spectrum of an LPG (Λ = 649 µm, 40 points) as a function of the refractive index of the external medium, in the range between 1.000 and 1.426.



Fig. 2. Transmission spectra of the same LPG comparing the attenuation dip for two different refractive indexes of the external medium: water (top) and a mixture of water and glycerine (bottom). The refractive index of the external medium is shown in the lower left corner of each graph.

Fig. 3 shows the peak position of the resonant band in the transmission spectrum of the same LPG when the fibre optic is immersed in a mixture of ethanol and commercial gasoline (Brazilian standards). The initial point in that graph corresponds to the legal proportion of 24% of ethanol in the mixture. Higher ethanol concentrations are obtained by the proper mixture of pure ethanol to the legal gasoline, tracking the alcohol proportion during the whole process. Using the pure ethanol as external medium is possible to obtain the point corresponding to the concentration of 100 %. The graph shows the sensitivity of the LPG when used as a sensor for the proportion of the mixture, based on the fact that the refractive index of a mixture of two liquids has a value depending on their respective volumetric concentration⁸. It can be seen on that figure that the sensor is useful to determine ethanol proportions (in volume) even higher than 60 %, although the region with greater sensitivity, where the slope is approximately linear, lies between 24 % and 49 % (see insert in Fig. 3). This region is particularly useful because most of the frauds and malpractices using higher concentration of ethanol are within such range (for higher concentrations the engine may not work properly, which attracts the attention of consumers). The best fitting slope in that linear region is 0.17 ± 0.02 nm/%. It is also important to notice that the absolute variation of the peak position of the resonance band is around 4.5 nm in that region, a dynamic range that assures the possibility of even better accuracy and resolution, when using LPG with narrower line widths. Another improvement that can be used to increase accuracy and resolution is the use of thermal annealed fibre optic to write the long period grating. It has been shown that fibres annealed by half an hour at 1050 °C can dramatically improve the resonance band in the transmission spectrum⁹ of LPG formed by the electrical arc process.



Fig. 3. Peak position of the resonance band in the transmission spectrum of the LPG as a function of ethanol proportion (in volume) between 24 and 100 %. The insert shows the region with higher sensitivity (24 and 49 %), that can be described by a linear relation with angular coefficient of 0.17 ± 0.02 nm/%.

3. CONCLUSION

We report the measurement of ethanol proportion when mixed with gasoline, using a Long Period Grating based sensor. Calibration of the process has been checked by the use of liquids with known refractive index. The average resolution was 0.0116 nm-1 when the refractive index varied from 1.333 to 1.426. For the ethanol measurements the average sensitivity for the wavelength shift of the resonance band is 0.17 ± 0.02 nm/%, for the region where the ethanol proportion changed from 24 to 49 % in the mixture with gasoline. The process can be used with other hydrogen – carbon products that are also used in the gasoline and that can suited to fraudulent uses.

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