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Experimental Difficulties with LPG Sensors Operating Close to the Phase Turning Points

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Abstract— In this paper we report on experiments aimed at the development of a highly responsive sensor based on long period gratings (LPGs) operating at high order cladding modes which exhibit a turn-around-point in their phase matching diagrams. These high order cladding modes are accessed by decreasing the period of the grating within standard optical fiber according to theoretical simulations. The sensors tested detect hydrogen through a Pd coating on the fiber cladding. As expected from simulations these sensors exhibit a near 8 fold increase in response to 1% hydrogen when operated near the phase turning point. This increase in response is, however, not selective to hydrogen and a combination of increased thermal noise and broadened resonance condition serves to decrease the signal to noise ratio such that any gain in theoretical sensitivity is counteracted by a loss in fidelity of the recovered (or recorded) signal. The highest sensitivity (shift of a LPG lossband) recorded was in the order of 470pm for an exposure to 1% H₂ at 70°C, which represented a shift of ~1/150 of the FWHM of the LPG loss band.

Index Terms— Gratings, Optical fibers, Microsensor, Hydrogen

I. INTRODUCTION

There has been increasing interest in recent years over the development of long period gratings (LPGs) as environmental sensors. In particular, an all optical hydrogen sensor for use in the presence of high electromagnetic fields in remote environments would be beneficial. To date, however, many of the reported fiber optic sensor designs have relied on either unusual, therefore expensive, fibers [1-4] or on modified (e.g. taper, etched etc.) standard fibers [1, 2, 5-8]. A number of these demonstrated sensor designs are based on palladium [1, 2, 5-10], exploiting the catalytic and reversible

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hydrogen adsorption properties of this metal. Palladium readily absorbs large volumes of hydrogen by catalytic dissociation of H₂ into atomic H which then migrates into the Pd lattice and resides at specific, interstitial, locations. This catalytic reaction takes place even at room temperature and is reversible; hence hydrogen establishes equilibrium in concentration between the lattice and the environment. When hydrogen takes up a space in the Pd lattice, the metal expands, straining the lattice constant, which affects the conductivity and this, in turn, changes the complex refractive index of the material [11].

An LPG is fundamentally the same structure as a fibre Bragg grating (FBG), a periodic change in the refractive index of the core. Within an FBG the light is coupled from the core mode into the counter-propagating core mode (i.e. reflected). While in an LPG the light is coupled into the co-propagating radially symmetric HE_{1,v} cladding modes through the phase matching condition (1) [12].

$$\lambda_v = \Lambda n_{ec} - n_{ev} \quad (1)$$

where the resonant coupling wavelength of the vth cladding mode (λ_v) is related to the grating period Λ and the effective refractive indices of the core (n_{ec}) and vth cladding mode (n_{ev}).

Thus LPGs enable the interaction of a core mode with the external environment surrounding an optical fiber via coupling to the cladding modes by the use of appropriately scaled and tailored refractive index perturbations [13, 14]. If the external environment is then made to exhibit a change in physical properties such as a change in refractive index on exposure to hydrogen, this will then affect the effective refractive index which the cladding mode and hence the spectral properties of the transmitted core mode. Hence, by coating the fiber with a thin layer of Pd the LPG becomes a hydrogen sensor as strain induced in the Pd lattice leads to a change in complex refractive index when exposed to hydrogen. Provided that the Pd layer is thin (< 1 μ m) and the Pd remains in the alpha phase the response due to pure strain will be insignificant compared to the response due to refractive index since the forces which such a thin Pd layer can transfer to the much stiffer fibre of significantly larger cross section.

II. MODE ORDER SENSITIVITY

The sensitivity of the LPG hydrogen sensor is driven by both Pd-H₂ dynamics and the phase matching conditions in (Eq. 1). For example, at an elevated temperature the thermo-

optic coefficients of the fiber will affect the effective refractive indices of the core and cladding modes. At the same time Pd absorbs hydrogen more quickly since the catalytic efficiency is increased. However, this increased reactivity driving the ingress of hydrogen into the lattice also leads to an increase in removal of hydrogen, thus the equilibrium concentrations will be lower at elevated temperatures. This then leads to a lowering of the overall change in the refractive index [2, 11] in Pd as a function of hydrogen concentration in the environment.

The Pd-H₂ dynamics are however fixed and other than layer thickness and temperature there is little that can be done to alter the performance of the sensor in this respect. There are a number of Pd alloys such as Pd-Ag and Pd-Cu which can be used to mitigate against contamination and cross sensitivities by CO and sulfides affecting the reaction dynamics of the Pd layer [15, 16]. This addition of any an alloy partners will always reduce the sensitivity. Since in our prospective end application high sensitivity is desired and as contaminants are not expected, we disregarded these more complex alloys. One of the critical benefits of the use of pure Pd is the near complete self-reversibility of the uptake of hydrogen at ambient conditions without external stimulation, whereas alloys of Pd are no longer fully reversible and hence will show hysteresis [17].

The parameter which is available for tuning and improving the sensitivity of such a system is the phase matching condition given in (1). Choosing the appropriate cladding modes to which the core mode is coupled to, within the wavelength range of interest, will strongly affect the sensitivity of the sensor [18].

As the effective refractive indices of the core and cladding are both dispersive, solving the phase matching conditions is

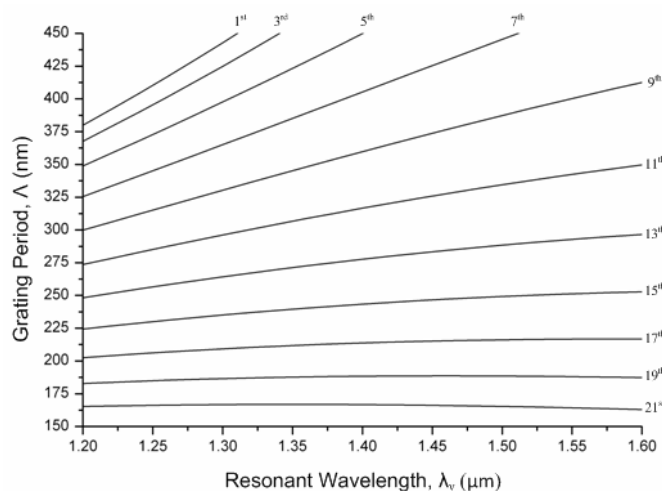


Fig. 1. Phase matching curves for the 1-21st order odd cladding modes for Corning SMF-28 fiber.

non-trivial. However, there are a number of suitable approaches reported in the literature [12-14, 19].

Figure 1 shows the phase matching curves for a standard telecoms (Corning[®] SMF-28[®]) fiber from 1.2 to 1.6 μm . By convention, even order HE_{1,v} cladding modes are not plotted

here in this graph since there is near-zero mode overlap with the core and hence near-zero coupling, however they are still being counted in our nomenclature.

The gradient of the curves gives an indication of the sensitivity of individual cladding modes. The lower order modes are steeper and hence are expected to be less sensitive since a small change in period results in a small change in coupling wavelength [20]. The gradient decreases with higher mode order until a phase matched turning point is achieved around the 17th-21st orders. These modes exhibit the shallowest gradient and hence are expected to exhibit the highest sensitivity.

A typical LPG with a period of $\sim 400 \mu\text{m}$ will couple to these higher order modes in the far infra-red (where the fiber does not guide and is highly absorbing). In order to shift the resonant wavelengths to a useful wavelength, i.e. in the spectral bands used by telecoms, it is necessary to either shift the effective refractive indices through the use of non-standard fiber, as has been demonstrated by Young Ho et. al. [4] or to decrease the period of the LPG by approximately one half. This is the approach we are using in this paper.

III. FABRICATION

Specifications for suitable low and high order LPGs were calculated though a computational model [21]. This model

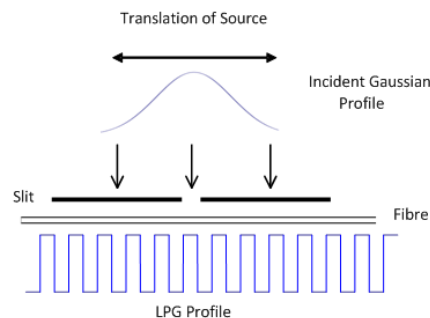


Fig. 2. Schematic of the LPG inscription system fixed slit 100 micron.

provides the period, strength and length of an ideal grating. Of these parameters, the period is critical for ensuring that the correct mode order is being coupled to, at the interrogation wavelength, while the strength and length determine the coupling efficiency.

Fabrication of the LPG elements was carried out by a point by point inscription method using hydrogen loaded SMF-28 fiber, illuminated with a 248 nm KrF excimer laser through slits of $\sim 1/2$ the width of the intended periodicity of the LPG, see Fig. 2. This slit allows individual high index periods to be inscribed into the LPG one at a time. Although the duty cycle of the grating should ideally be 50/50 this is not always the case due to erosion of the slit under high laser intensities. However, this has not been seen to cause detrimental effects and can be compensated by a marginal change in grating length.

The LPG is monitored in transmission during the inscription process. The length of the grating can therefore be increased until the desired transmission spectrum is achieved, although

the determination of the eventual LPG characteristics are complicated by the post-processing of the fiber; annealing and Pd coating. Annealing has the effect of partially bleaching the grating making it necessary to deliberately over inscribe the grating during fabrication. This process is, however, repeatable and so can be compensated through a trial process. The Pd coating induces both a change in the coupling efficiency, i.e. strength of the loss band, - and in the resonant wavelength.

An idealized spectrum of an LPG, post palladium coating can be modeled with a metal jacket and the idealized post

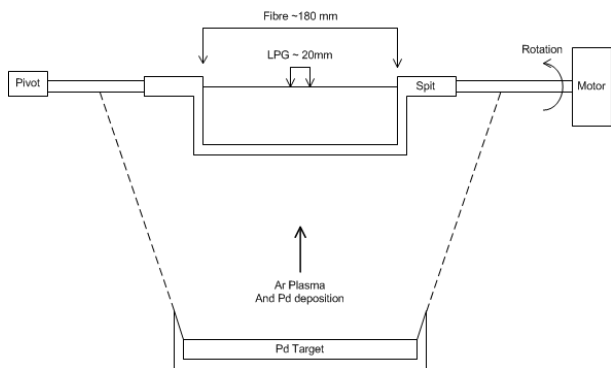


Fig. 3. Schematic of the RF sputter coating of an LPG with Pd

annealing spectrum can be calculated based on the mathematical framework presented in [21]. The fibers presented in this paper have a UV induced refractive index contrast of typically 3×10^{-4} refractive index units (RIU). Typical lengths are in the order of ~ 24 mm and the LPGs have been annealed to 200°C over a period of ~ 2 h.

The Pd deposition process was carried out by an RF sputter coating system in a low pressure Ar atmosphere ($\sim 9 \times 10^{-4}$ mbar) in an Edwards box coater unit, see Fig. 3. RF-Sputter coating produces smooth homogeneous surfaces with an essentially cold deposition process (care must be taken not to further anneal the grating) [22]. The thickness of the Pd coating has to be chosen with care and is subject to a range of potentially contradictory requirements. Very thin, < 20 nm, films have an uncertain complex refractive index and might not form a complete homogeneous coating, but rather an island type coating, where the deposited Pd migrates by up to a μm over the surface to form small islands of Pd. For thin films of < 90 nm the refractive index of the film is dependent on the thickness and is hard to predict. Excessively thick films, in excess of 1 to a few μm , will respond only very slowly to hydrogen since it is necessary for the interstitial hydrogen to fill the Pd “sponge” from the air/Pd surface, as only the interface between the fiber and the inner Pd surface will be probed by the cladding modes. Hence, an intermediate thickness of 100 nm was chosen for all Pd coated LPGs within this paper. Homogeneous coating over the full circumference of the fibers was carried out by rotating the fibers at a fixed distance of 10 cm above the RF source. A deposition rate of 1 nm s^{-1} was used. A witness slide was coated simultaneously, and also under rotation, to confirm the deposition thickness [22].

IV. INTERROGATION METHOD

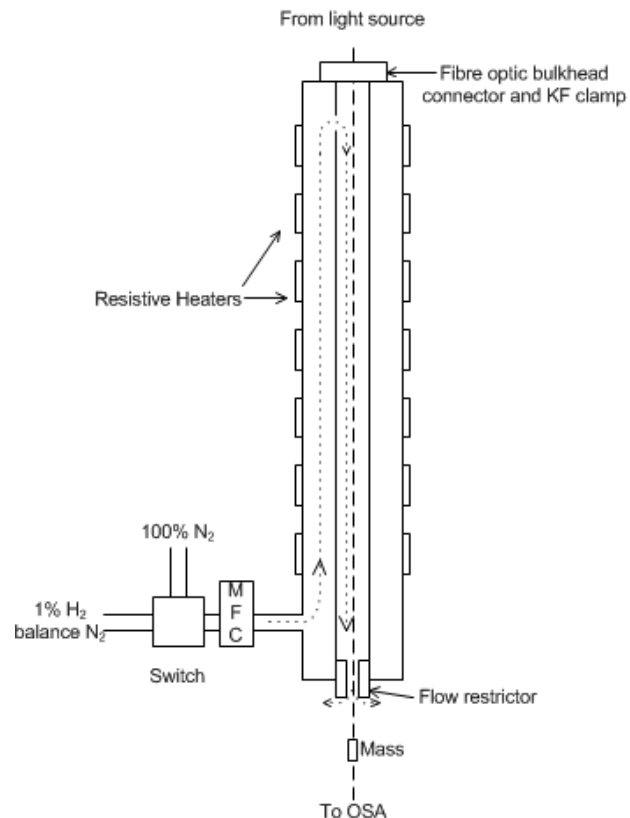


Fig. 4. LPG interrogation Rig diagram add strain weight

Interrogation of the LPGs was carried out using an Advantest Q8384 Optical Spectrum analyzer controlled via LabVIEW. Single Transmission scans with resolution 0.5 nm over a range of 100 nm were taken using a fiber coupled tungsten lamp for broadband illumination as no single superluminescent LED can provide sufficient bandwidth and combined sources over equivalent spectral widths are prohibitively expensive. A Gaussian peak was fitted to the loss band to retrieve the resonant wavelength.

Figure 4 shows the experimental setup used for exposing the LPGs to a known concentration of hydrogen. The LPGs were mounted hanging vertically in a tube and slightly tensioned by an attached weight at the bottom (typ. 20 g). This ensured that the fibers were tensioned at a constant and temperature independent force. Gas was supplied through a mass flow controller (Aalborg GFC171) set to flow at 100 sccm and an electronic switch, tied into LabVIEW, selected between 100% N_2 and 1% H_2 in N_2 . The gas is fed through a double walled chamber where it is heated to the desired operating temperature between 25 and 70°C before it enters the region where the LPG is located. This ensures temperature stability of $\pm 1^\circ\text{C}$ which helps to reduce thermal noise and eliminates uncertainty resulting from the temperature dependence of the Pd- H_2 absorption. The raw spectral data was fitted to a Gaussian profile to determine the position of peak loss - the resonance position. Plots of time against LPG resonance position and hydrogen concentration are therefore derived.

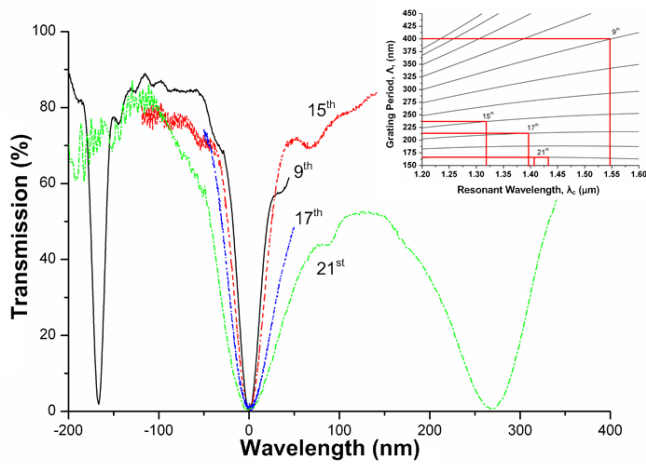


Fig. 5. Overlaid plots of the transmission spectra of the 9th, 15th, 17th and 21st order modes. Plots have been centered about the resonant coupling wavelength and the transmission normalized to this point for clarity. Inset indicates actual resonance positions and grating periods on phase matching curves. The 9th order mode shows two separate mode resonances while the 21st order mode demonstrates double resonance.

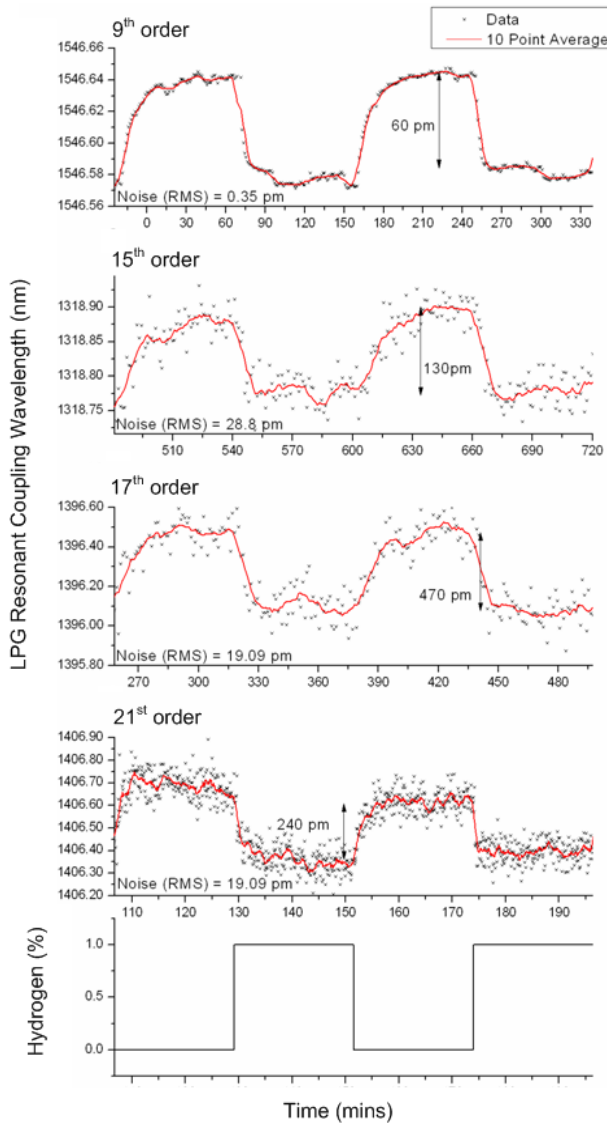


Fig. 6. Plots of hydrogen response of the 9th, 15th, 17th and 21st order modes with the RMS noise labeled. Lower plot indicates hydrogen exposure.

V. RESULTS AND DISCUSSION

Figure 5 shows a plot of the 9th, 15th, 17th and 21st order mode resonances for four separate LPGs. It should be noted that the resonant wavelengths of each of these LPGs are not the same, but have been shifted and stacked for convenience to their central wavelength. Of particular note is the increase in resonance width due to the flattening of the phase matching conditions, and the appearance of a double valley spectrum for 21st order LPG, which appears when using a periodicity close (within a μm) to the phase matching turning point. This figure clearly illustrates the difficulty in determining the centroid position of the LPG in view of the increasing width of the spectral loss band for higher mode orders. It is also worth noting that the higher order modes, particularly with hydrogen loaded fibers, are inherently more challenging to manufacture with accuracy. Since the sensitivity of these modes is much higher, determining the correct stop position, particularly before annealing, is extremely challenging.

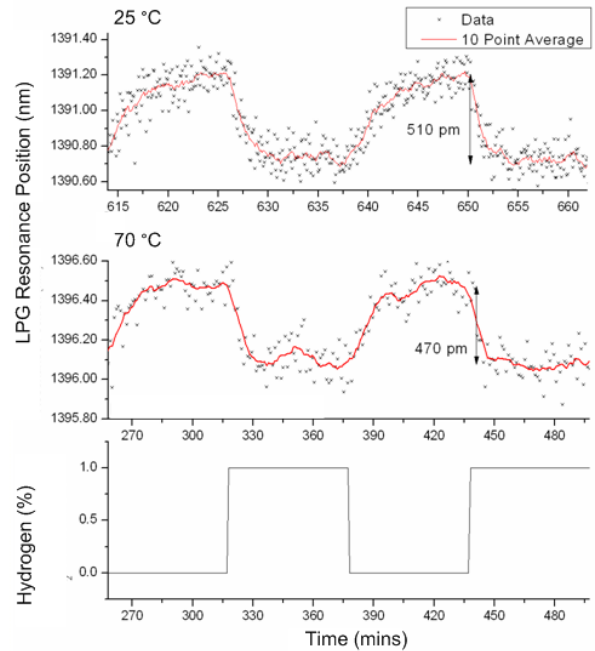


Fig. 7. Temperature responses of a 17th order LPG at 25 and 70 °C.

The results in Fig. 6 show the response of a sensor based on the 9th order mode. Here a sensitivity of 60 pm to 1% H₂ with a RMS noise of 0.35 pm is demonstrated. Figure 6 also shows equivalent plots for the 15th, 17th and 21st order modes giving responses of 130, 470, 240 pm and RMS noise of 28.8, 19.09, 19.09 pm, respectively. The increased noise at the 15th order is the result of interrogating the LPG at around 1320 nm, which is within the range of OH absorption lines present in fused silica fibers.

Figure 7 shows the effect of temperature on the LPG-Pd system. There are two separate effects present. A shift in temperature gives a D.C. offset of 5.2 nm to the resonant wavelength of the LPG by altering the effective refractive indices via the thermo-optic coefficient. There is also a change in the Pd H₂ absorption dynamic. At lower temperature there

is an increase in the scale of response (i.e. sensitivity) but a decrease in the rate of response.

By increasing the mode order towards the phase turning point, the absolute response of the LPG-Pd system has increased by almost 8 times (from 60 to 470pm at 70 °C). This has, however, been accompanied by an even larger, factor of 54, increase in the noise of the measurement (0.35 to 19.09 pm). This signifies a significant worsening in the signal to noise ratio of the system with increasing mode order. The low signal to noise in turn results in an increase in the detection limit of almost an order of magnitude from 175 ppm to 1220 ppm [23,24].

This decrease in signal to noise ratio can be attributed to a combination of two factors. Firstly, as the mode order increases, the spectral bandwidth of the mode coupling conditions increases, while the coupling coefficient (depth) decreases. This results in a shallower and wider resonance which, in the case of the 21st order is approximately 70nm at FWHM. A shift of 470 pm of the 70 nm wide peak represents a peak shift of 1/150th of the FWHM and even small perturbations in the overall peak shape, the spectral power of the illumination source and variations in the transmittance of a coiled and flexible fiber optic down lead [25] can affect the accuracy of the measured peak position. At the same time, the increase in mode order has also increased the sensitivity to other external stimuli, particularly temperature which would require even more accurate temperature compensation.

VI. CONCLUSION

Although the increase in absolute response opens the possibility of measuring hydrogen using lower resolution, more cost effective equipment, the end result is that our high mode order LPG sensor does not exhibit an improvement in sensitivity in real terms due to the decrease in signal to noise ratio. Higher sensitivities are therefore dependent on improving the signal to noise ratio. There are a number of potential approaches which may be used in this case including; isolating the sensor from unwanted stimuli; compensating for temperature variations; improving the speed of data acquisition or improving the accuracy of the system determining the loss band position. However, the spectral dependency of bend losses in single mode fibers will become a limiting factor for such wide lossbands. Alternative sensor designs which provide a high response while maintaining a narrow resonance, e.g. tilted gratings [26], may provide a more attractive, practical, option.

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