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# A fibre optic humidity sensor based on a long-period fibre grating coated with a thin film of SiO<sub>2</sub> nanospheres

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## Abstract

A novel sensing configuration for measuring humidity based on a long-period fibre grating coated with a thin film of silica nanospheres is proposed. The polymeric overlay is deposited on the grating using the electrostatic self-assembly technique. This thin film changes its optical properties when exposed to different humidity levels that translate into a shift of the resonance wavelength of the fibre grating. Wavelength shifts up to 12 nm in a relative humidity range from 20% to 80% are reported, and it is further demonstrated that such humidity sensitivity has negligible thermal dependence.

**Keywords:** humidity sensor, long-period fibre grating, nanospheres, electrostatic self-assembly

## 1. Introduction

Humidity control is a critical parameter in a large number of economic activities, such as medical procedures or food processing. Therefore, there is a need for devices to measure humidity, the use of conventional electrical-based sensors being common. However, in many situations, it would be highly advantageous to have optical sensors for several reasons, which include safe operation in hazardous environments, immunity to electromagnetic noise and the possibility of performing remote and multiplexed measurements when optical fibre sensors are considered.

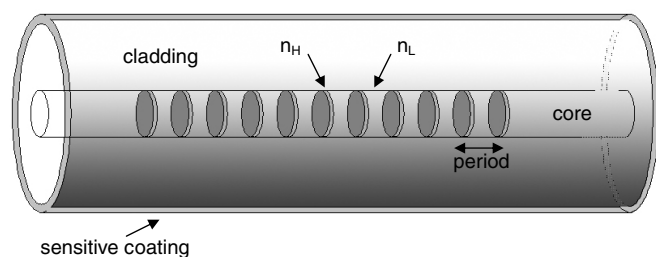
To exploit such technical advantages, several optical fibre based sensing configurations for measuring humidity have been reported in the literature. It is the case of using side-polished fibre [1], U-bend [2], nano-Fabry–Perot cavities [3] or tapers [4]. Also, the use of different techniques has been demonstrated to enhance the measurement sensitivity, one of the most widespread being based on the deposition of polymeric sensing layers [1, 5–7].

Taking advantage of the hydrophilic nature of silica nanospheres, we can measure humidity-induced shifts in the optical properties of the material when it is structured as a thin film. In this type of sensitive nanofilm, the coating

refractive index has a strong dependence on the number of water molecules in the interstitial gaps within the nanospheres. Given the small intrinsic dimensions of the overlay, this kind of sensor is characterized by a fast response time.

Nanospheres have been widely used in other applications [8]. Taking advantage of the hydrophilic nature that the material exhibits, one can induce shifts in its optical properties. In this type of sensitive nanofilm the coating's refractive index changes when the number of water molecules in the interstitial gaps among the nanospheres increases or decreases.

Sensors using long-period fibre grating (LPG) [9, 10] technology have been widely researched considering their intrinsic interaction with the surrounding medium. Indeed, the dependence of the grating resonant wavelength on the external environment can be used to detect refractive index changes. This property can be further enhanced through the deposition of thin films on the fibre surface, since LPG promotes cladding mode coupling that interacts with the film inducing resonant wavelength shifts in the presence of environmental changes. There are many LPG-based sensing applications that exploit such favourable properties, ranging from pH measurement [11] to refractometers [12] or even detection of chemical substances [13].



**Figure 1.** The humidity sensor structure based on a LPG.

In this work, an optical fibre humidity sensor based on a novel film of silica nanospheres deposited over the cladding of an LPG is presented. Although the sensitive overlay has an index of refraction far from the refractive index of the fibre itself, which is the optimum situation for sensitivity optimization [14], yet a wavelength shift of  $\sim 12$  nm was obtained for changes in the relative humidity ranging from 20% to 80%.

## 2. Experimental details

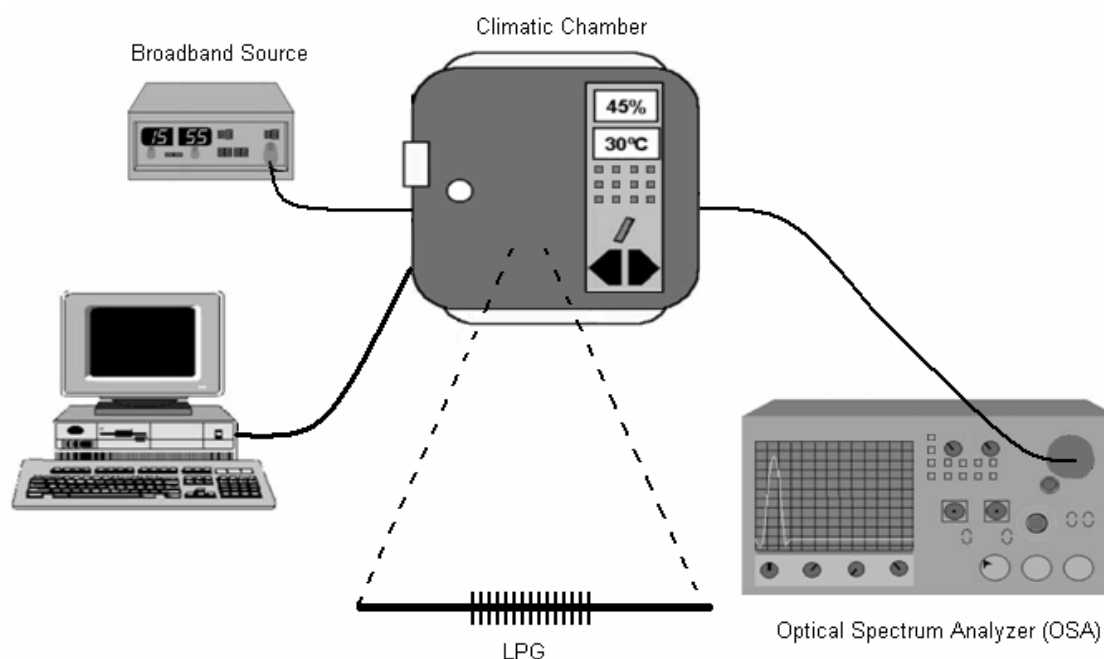
The humidity sensor structure is shown in figure 1. The LPG was coated with a sensitive layer using electrostatic self-assembly (ESA) [15]. In this work, the materials employed were poly(allylamine hydrochloride) (PAH), poly(sodium 4-styrenesulfonate) (PSS) and LUDOX<sup>®</sup> SM-30 SiO<sub>2</sub>-water colloidal suspension (all from Sigma-Aldrich). The PAH acted as a polycation, and the PSS and the SM-30 were the anionic species. Aqueous diluted solutions of PAH, PSS and SM-30 were prepared with concentrations of 10 mM for PAH and PSS relative to the polymer repetitive unit, and 1%wt for the

SM-30. The pH value of all solutions was adjusted to 4.0 just by adding a few drops of HCl or NaOH. The LPG used in this experiment had a length of  $\sim 25$  mm and a period of  $240 \mu\text{m}$ . The LPG substrate was thoroughly cleaned by oxygen plasma etching, and afterwards it was immersed into the solutions. Firstly, in order to improve the adhesion of the subsequent nanostructure, four bilayers of (PAH/PSS) were deposited, using immersion cycles of 2 min. Afterwards, 14 bilayers of (PAH/SM-30) were added, and in this case the immersion time was increased to 15 min. Throughout the fabrication process and after each immersion step, a 3 min rinsing bath in gently stirred ultrapure water was carried out. All the fabrication process was carried out using a dipping robot (R&K GmbH) in order to minimize experimental errors.

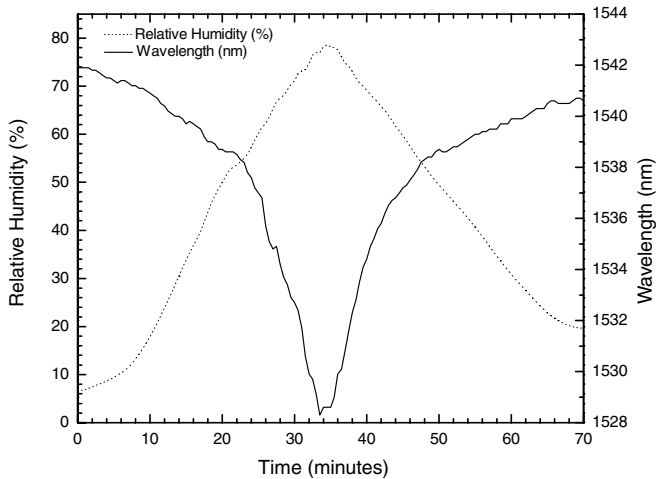
Figure 2 shows the experimental setup used to characterize the sensor. The transmission spectrum of the LPG was acquired through a broadband source and an optical spectrum analyzer. Several experiments were implemented changing the relative humidity from 20% to 80% at distinct temperatures within a range from 10 °C to 40 °C. These experiments were made using a climatic chamber (ANGELANTONI INDUSTRIE, model CH250) that allows humidity and temperature to be changed under controlled conditions.

## 3. Results and discussion

The wavelength shift induced by a humidity cycle performed within the range from 20% to 80% at a constant temperature of 25 °C is shown in figure 3. The humidity variation is represented with a dashed line, while the wavelength shift of the absorption peak of the LPG is represented by a straight line. Note that, when humidity increases, the resonance



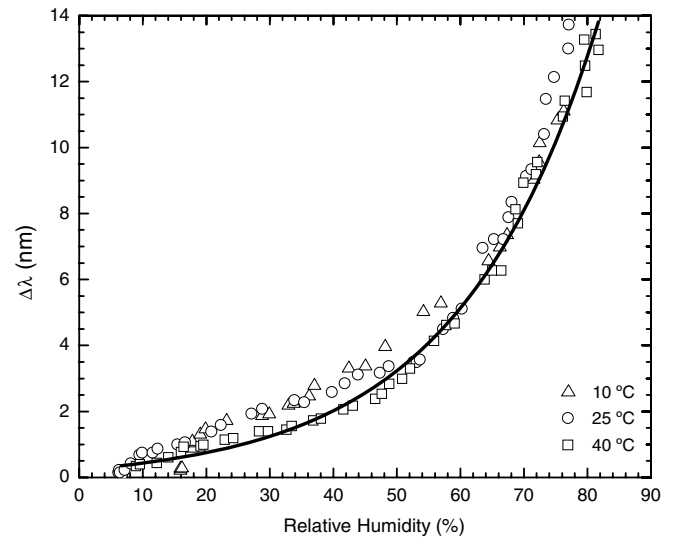
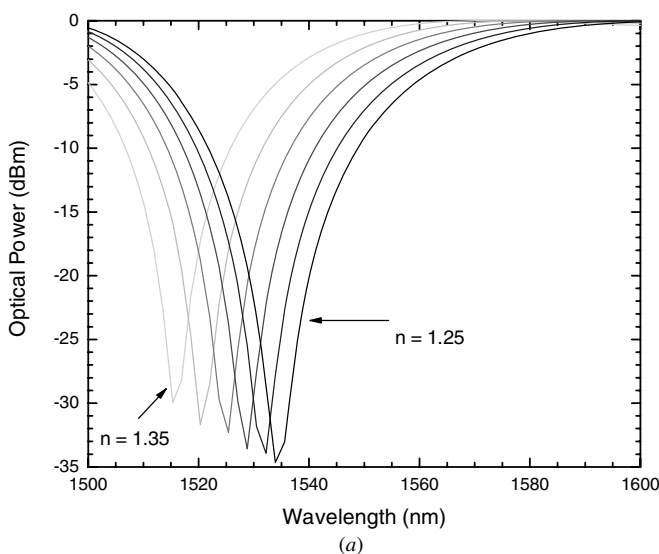
**Figure 2.** Experimental setup.



**Figure 3.** Resonance wavelength shift for different relative humidity levels.

wavelength peak shifts to lower wavelengths. This effect is due to changes accomplished in the refractive index of the film of SiO<sub>2</sub> nanospheres. A similar dependence is observed by reproducing the same humidity cycle at different temperatures.

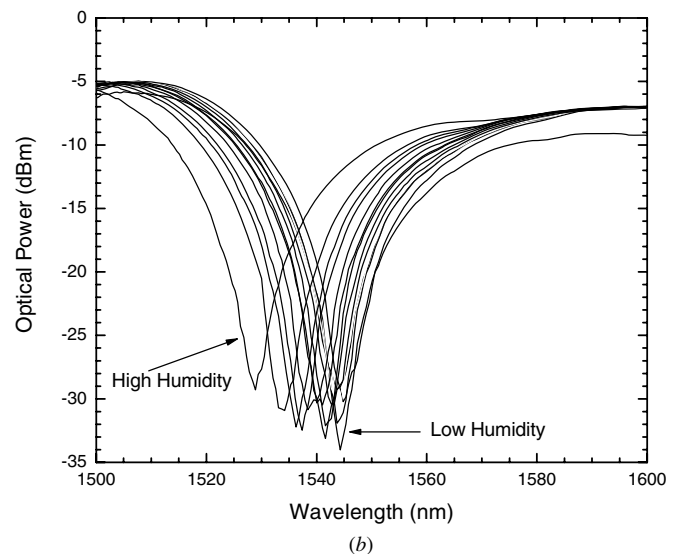
Theoretical simulations were made to understand the behaviour of the LPG with changes of the external refractive index [16]. Figure 4(a) shows some simulated LPG spectra considering a coating with a refractive index of  $\sim 1.3 + 0.003i$  and a thickness of 350 nm. The simulation was made for an overlay refractive index ranging from 1.25 to 1.35, corresponding to the values of the refractive index in the relative humidity operating range. The steps involved in the film fabrication indicate that a film thickness close to the value used in the simulation can be expected. Figure 4(b) shows the experimental results obtained for different relative humidity levels.



**Figure 5.** Resonance wavelength shift of the LPG versus relative humidity levels for three different temperature values (constant offsets were removed).

The resonant wavelength shifts observed in the experimental data are consistent with the theoretical results, since the rise of humidity implies an increase in the refractive index. For high humidity levels, the interstitial spaces in the film formerly filled with air are now filled with water molecules, which cause the refractive index to increase. The attenuation observed in the resonant peak in figure 4 is due to the absorption of the polymeric overlay, as demonstrated in previous works [17].

With the obtained spectra, the dependence of the resonance wavelength shift on the relative humidity can be plotted, as given in figure 5. The present data were obtained throughout several humidity cycles performed at



**Figure 4.** LPG spectra: (a) theoretical simulation for different refractive index values of the overlay; (b) experimental results for different relative humidity levels.

different temperatures. As expectable, the sensing head is also sensitive to temperature, showing a coefficient of  $\sim 100 \text{ pm } ^\circ\text{C}^{-1}$ . However, figure 5 shows that, after subtracting the temperature-induced offset, the wavelength shift on humidity fits the same dependence regardless of temperature, given by the following exponential growth:

$$\Delta\lambda = 3.85 \times 10^{-1} \exp\left(\frac{R_H}{2.11 \times 10^1}\right) - 1.26 \times 10^{-1},$$

where  $R_H$  is the relative humidity, and  $\Delta\lambda$  is the wavelength shift. This is the curve which best fits the obtained experimental results in the operation range from 20 to 80%. In a more extended range or if working at another working point, then this analytical expression must be changed. More details about the evolution of the resonance peak as a function of the index of refraction can be found in [18].

This result demonstrates that temperature cross-sensitivity can be effectively compensated by implementing standard thermal compensation techniques.

This result demonstrates that the temperature does not change the sensor calibration dependence, inducing only an offset. Therefore, it is straightforward to compensate the temperature-induced effects just by reading the sensing head temperature, which can be done in different ways, using, for example, an in-line fibre Bragg grating.

Due to large inertia of the experimental setup, it was not possible to determine the response time of the implemented humidity sensor. Additional experimental work is being carried out for this purpose. Preliminary results indicate that this response time is in the order of a few milliseconds.

#### 4. Conclusions

A novel humidity sensor based on a LPG coated with a thin film of  $\text{SiO}_2$  nanospheres was implemented and characterized. The obtained results demonstrate an exponential dependence of the resonance wavelength shift on humidity, but not on temperature.

#### Acknowledgments

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