

## Characterization of optical fiber long period grating refractometer with nanocoating

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### ARTICLE INFO

#### Article history:

Received 16 June 2010

Received in revised form 15 October 2010

Accepted 22 October 2010

Available online 10 November 2010

#### Keywords:

Optical fiber sensor

LPG sensor

Nanocoating deposition

### ABSTRACT

In this work is studied the response of optical fiber long period grating (LPG) to changes of the refractive index of the external media relatively to variations of wavelength and in transmission. The response of the LPG to refractive index greater and lesser than to cladding is investigated. A nanolayer was deposited onto the fiber to increase the sensitivity of the LPG to refractive index of the external media higher than cladding. The film modifies the rates of effective modes of cladding, thus improving the response of the changes in the refractive index of the external media higher than that in the refractive index of the cladding ( $n_{cl} \approx 1.457$ ). The Langmuir–Blodgett technique was used for the deposition of the nanolayer.

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### 1. Introduction

Optical fiber sensors based on long period gratings (LPGs) have been widely studied for the last years. They have been used in filtering applications such as gain-flattening and band-rejection, and for sensing of strain and temperature [1–4]. A feature of LPGs is their sensitivity to the index of refraction of the material surrounding the cladding glass in the grating region. This arises from the dependence of the attenuation band wavelengths on the effective index of refraction of the cladding modes, which depends on the index of refraction of the surrounding environment, allowing to use LPGs as sensing element devices in analytical measurements of the chemical and environmental fields.

The optical fiber refractometers have certain advantages relatively to conventional devices such as, resistive, piezoelectric and solid state: immunity to electromagnetic radiation, small dimensions and low weight (length between 2 and 20 mm and diameter of 125  $\mu\text{m}$ ), flexibility, high melting temperature, electrical passivity, high sensibility, high bandwidth, capability of monitorization of several parameters of interest simultaneously, multiplexing (several sensors can be written in the same fiber which allows measurements spatially distributed), non-electric and non-conductor device, resistant to corrosive environments, long-distance transmission, possibility of real-time monitoring, low reactivity of silica

to the external media and chemically inert to water and chemicals [5].

Long-period gratings can be considered a special class of fiber Bragg gratings in which the period of the index modulation is such that it satisfies a phase matching condition between the fundamental core mode and a forward propagating cladding mode of an optical fiber. For a LPG, a periodic modulation of the index of refraction in the fiber core has typically a period in the region from 100  $\mu\text{m}$  to 1000  $\mu\text{m}$  and a length of a few cm, which is induced in the fiber using different processes: UV irradiation source, arc-electric discharge, period etching,  $\text{CO}_2$  irradiation source and mechanical process.

Vengsarkar et al. [6] reported the principle of operation and some characteristics of LPG. The LPG operates by coupling radiation in which the fundamental guided mode is perturbed by the presence of the grating in the fiber core. The difference between the propagation constant of the guided mode and the phase-matching vector of the grating equals the propagation constant of one or more cladding modes at appropriate wavelengths which correspond a cladding mode order. The phase matching condition between the fundamental mode and the forward propagating cladding mode is given by equation:

$$\lambda_m = (n_{co} - n_{cl}^m) \Lambda$$

where  $\lambda_m$  is the peak wavelength of the resonance band between the core mode and the cladding mode,  $n_{co}$  and  $n_{cl}^m$  are the effective refractive indexes of the core mode and of the  $m$ th order cladding mode, respectively, and  $\Lambda$  is the grating pitch.

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The LPG sensitivity to environmental parameters is influenced by the grating pitch and by the order of the cladding mode to which coupling takes place. In particular, the surrounding sensitivity refractive index arises from the dependence of the phase matching condition upon the effective refractive index of the cladding modes, which are dependent on the difference between cladding and surrounding refractive indexes [7]. As the external index changes from the unit to near cladding index, the principal effect is a blue-shift of the center wavelengths of the bands which is particularly pronounced in the longest wavelength bands [7]. In region of the cladding index an abrupt change in the spectral characteristics is observed. At this point the cladding modes would be expected no longer being discrete guided modes and as seen the coupling spectrum spreads and the coupling nearly disappears for the highest order modes. For external index higher than the cladding, coupling to cladding mode structure reappears, with the highest order bands increasing in strength with increasing index. The shortest wavelength coupling bands remain visible over the full range of indexes [7].

For values of refractive index higher than 1.46 the sensitivity of the LPG to the refractive index of the external media is very low, having minimum displacements of wavelength; however the sensitivity of the LPG to the refractive index of the external media can be increased by the deposition of a film of organic material on the fiber.

The idea to coat LPG with higher refractive index layers was, firstly, proposed by Tatam and coworkers [8]. The experimental results showed that a high refractive index (HRI) overlay induces strong changes on spectral properties of a standard LPG, and Del Villar et al. [9] proposed a comprehensive theoretical and numerical investigation demonstrating that HRI coatings were able to favor the transition between cladding guided modes in overlay guided modes, causing a strong re-distribution of the cladding modes.

The response of the LPG is modified according to the refractive index and the thickness of the film. Thus, to identify composites whose next refractive index is higher than 1.47 this type of sensor can be used. The deposition of the film modifies the effective indexes in the coat attenuation bands, thus improving the response of the grating to variations of the refractive index of the external media higher than that of the refractive index of the cladding. Some authors had used the Langmuir–Blodgett (LB) technique to produce the nanolayer [10,11]. An LB film can be deposited by passing a solid substrate such as a glass slide or optical fiber vertically through a condensed Langmuir layer. The most common multilayer is the Y-type, which is produced when the monolayer deposits onto the solid substrate in both up and down directions.

Molecules constituting the LB film are usually amphiphilic, i.e. they have hydrophobic and hydrophilic tail or head groups. The technique is based on the fabrication of organic monolayer films, which are firstly oriented on a subphase and subsequently transferred, layer by layer, onto a solid surface at room temperature and molecular specific pressure. This offers a high resolution control over the film thickness, and so is ideal for waveguide applications.

In summary, in this work a study of the response of LPG to variations of the refractive index of the external media was made. A nanolayer of tricosenoic acid, using the LB technique, was deposited onto the fiber to increase the sensitivity of the LPG to refractive indexes of the external media higher than that of cladding. The response of the LPG to refractive indexes greater and lesser than the cladding was studied.

## 2. Materials and methods

The LPG was written using the electric-arc technique described by Rego et al. [12] in standard monomode optical fibers. The period

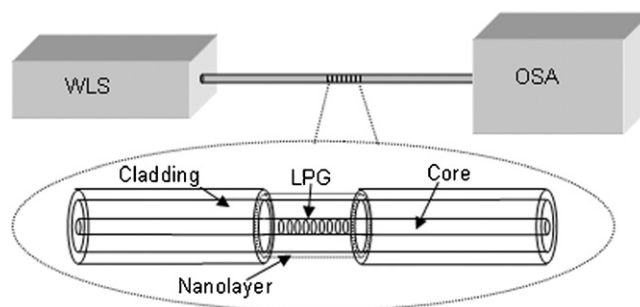


Fig. 1. Schematic representation of the interrogation system of LPG.

of the refractive index modulation was 395  $\mu\text{m}$ , a value chosen to produce a resonance wavelength at approximately 1550 nm. This resonance wavelength corresponds to the 6th order cladding mode.

The experimental setup used to characterize the sensing element constituted by the LPG with a LB nanolayer is shown in Fig. 1. A broadband white light source (WLS) illuminates the sensing element. From the transmitted broadband spectrum, the refractive index can be measured from the spectral shift of the LPG attenuation band. This shift was monitored using an optical spectrum analyzer (OSA).

The refractive index of the samples for analysis was previously measured with an ABBE refractometer.

For calibration, the sensing head was immersed in different external refractive indexes. For this, the sensor is inserted into a homemade glass cell with four openings, two of them used to pass the optical fiber with the LPG, and the other two to fill and to drain the samples. With the LPG inserted into the cell, the fiber ends are immobilized to avoid fiber-bending interference on the sensor response (Fig. 2).

This first calibration was without nanocoating deposition. To characterize the behavior of the grating the transmission values were plotted versus the values of central wavelength of the transmission band. In order to increase the sensitivity of the grating to refractive indexes higher than cladding, a film with refractive index higher than cladding was deposited.

A monolayer of the material is formed by applying the molecules to the surface of a subphase in the form of a solution. The solvent is allowed to evaporate, leaving the molecules dispersed across the water surface and oriented with the hydrophobic part upwards and the hydrophilic part in the water, producing a floating monolayer. Reducing the surface area by means of a moving barrier, the molecules begin to repel one another, modifying the surface pressure. The change in surface pressure may be monitored to produce a surface pressure isotherm. The

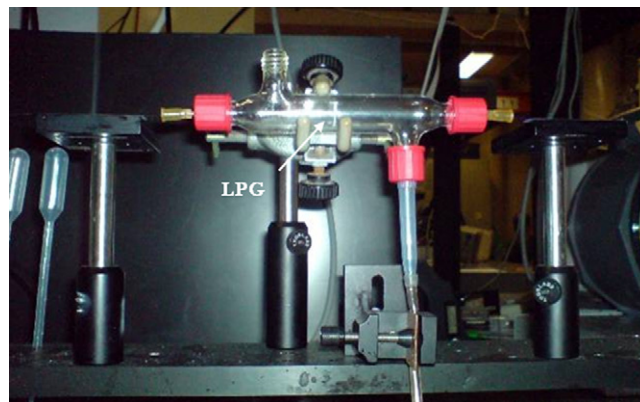


Fig. 2. Representation of a glass cell with a LPG.

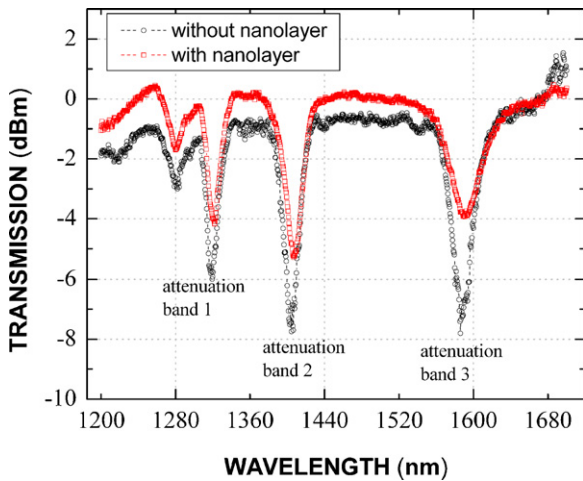


Fig. 3. Transmission spectrum of LPG with air as external medium.

isotherm has three distinct phases; gas, condensed liquid and solid.

The material deposited onto the fibers was tricosenoic acid ( $\text{CH}_2\text{CH}(\text{CH}_2)_{20}\text{CO}_2\text{H}$ ). This material has a molecular length of 2.7 nm, and refractive index 1.57 at 633 nm. The tricosenoic acid was spread from dilute chloroform solution ( $0.1 \text{ mg mL}^{-1}$ ) onto the pure water subphase (conductivity  $18 \text{ M}\Omega \text{ cm}$ ) of one compartment of a Nima Technology LB trough. Deposition was achieved at a surface pressure of  $30 \text{ mN m}^{-1}$  and a transfer rate of  $8 \text{ mm min}^{-1}$ . The fiber containing the LPG was positioned vertically so its long axis was aligned with the dipping direction and was alternately raised and lowered through the floating monolayer at the air–water interface. This procedure gave a Y-type structure in which the amphiphilic molecules were packed head to head and tail to tail.

Multiple passes through the film of molecular layer thickness 2.7 nm were carried out to prepare LPG with nanolayers of different thicknesses.

### 3. Results and discussion

The deposition of the tricosenoic acid onto LPG induces a modification of the distribution of the cladding modes leading to a wavelength shift of the attenuation bands combined with amplitude changes. Fig. 3 shows the changes induced in the transmission spectrum with and without nanolayer with air as external medium.

Since the tricosenoic acid film refractive index is 1.57 and the layer thickness is less than 110 nm a displacement of all attenuation bands is expected for the blue-shift wavelengths [9]. After the deposition, the LPG transmission spectra with air as external medium showed a blue-shift wavelength of 2 nm in the  $\text{LP}_{14}$ , 3.5 nm in the  $\text{LP}_{15}$ , and 4 nm in the  $\text{LP}_{16}$ .

The changes of the LPG transmission spectrum, for  $\text{LP}_{16}$  without nanolayer, with the variations in the refractive index of the external medium are shown in Fig. 4.

The displacement for lower wavelengths is verified until the value of the refractive index of the external attenuation band to the LPG is similar to the refractive index of the cladding of the optical fiber ( $n_{cl} \approx 1.4570$ ). After this value a displacement is observed for higher wavelengths and a marked attenuation of the spectrum. The transmission band has its maximum attenuation in this region. For refractive indexes close to the surrounding refractive index of 1.4725, it is observed an increase of the transmission efficiency.

Figs. 5–7 show the variation of the wavelength with the refractive index, for the  $\text{LP}_{14}$ ,  $\text{LP}_{15}$  and  $\text{LP}_{16}$ , respectively, for the LPG with and without nanolayer deposition. For the results with-

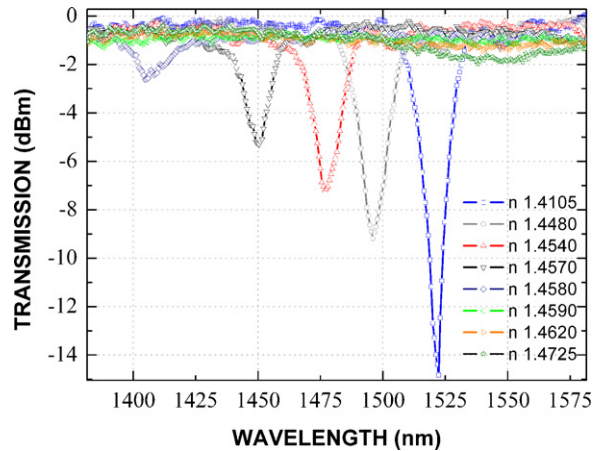


Fig. 4. Changes of  $\text{LP}_{16}$  of the LPG transmission spectrum with the external media.

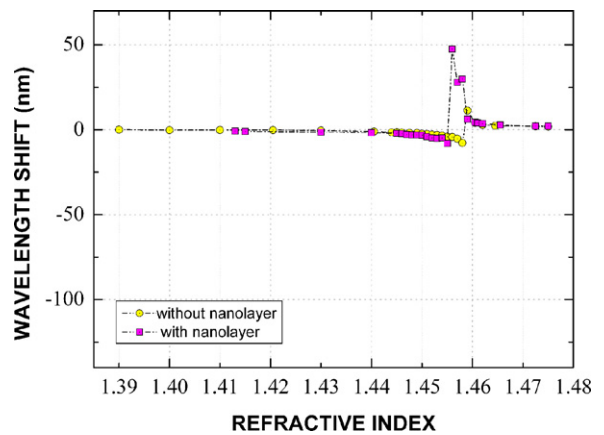


Fig. 5. Experimental values of the variation of the wavelength of the LPG with the surrounding refractive index for  $\text{LP}_{14}$ .

out nanolayer the sensitivity to variations in the external index increases with the order of the mode. When the surrounding refractive index is greater than the cladding, the sensitivity of LPG is low.

With a nanolayer of 110 nm, as in the case without film, the sensitivity to external index increases with increasing order of the mode. The major difference when compared with the case without film is an increase of the sensitivity for the region where the index outside is higher than the cladding. For example, concern-

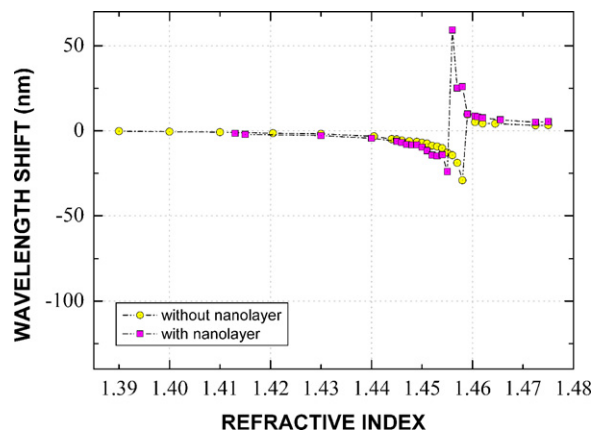


Fig. 6. Experimental values of the variation of the wavelength of the LPG with the surrounding refractive index for  $\text{LP}_{15}$ .

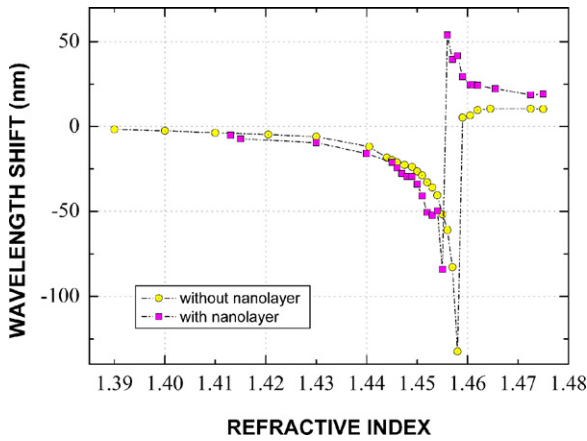


Fig. 7. Experimental values of the variation of the wavelength of the LPG with the surrounding refractive index for LP<sub>16</sub>.

ing the LP<sub>16</sub>, the value of refractive index of 1.46 the wavelength shift observed without nanolayer is 6.5 nm and with nanolayer is 24.7 nm.

In the region of the cladding index, an abrupt change in the spectral characteristics was observed. For external refractive index higher than the cladding, coupling to cladding mode structure reappears, with the highest order bands increasing in strength with increasing index. The shortest wavelength coupling bands remain visible over the full range of indexes.

For values of refractive index higher than 1.46 the sensitivity of the LPG to the refractive index of the external media is very low, having minimum displacements of wavelength; however the sensitivity of the LPG to the refractive index of the external media can be increased by the deposition of a film of organic material onto the fiber.

Fig. 8 shows a detail of the region of LP<sub>16</sub>, of refractive index higher than cladding. For higher refractive index, with the LPG without nanolayer the wavelength shift was reduced (with maximum variation of wavelength of the order of 0.05 nm between the samples). This LPG shows an enhancement of variation to the refractive index of external media due to the thickness of the nanolayer (110 nm). Thus the response of LPG is modified according the refractive index of the film.

As seen in Figs. 9–11, the sensitivity to refractive index manifests itself as a change in the minimum transmission value of the attenuation bands. The highest sensitivity is shown by the higher order

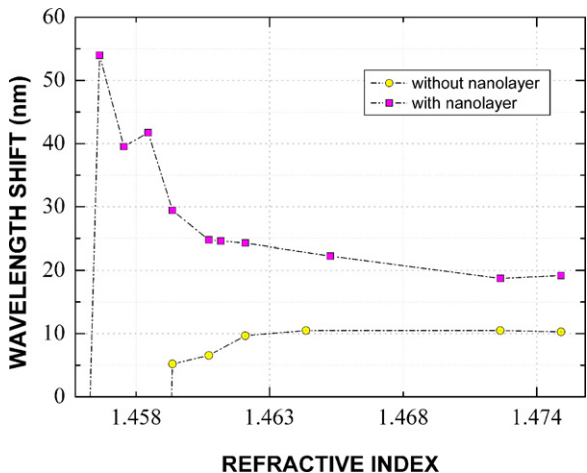


Fig. 8. Experimental values of the variation of wavelength with the surrounding refractive index with and without a nanolayer between 1.456 and 1.476.

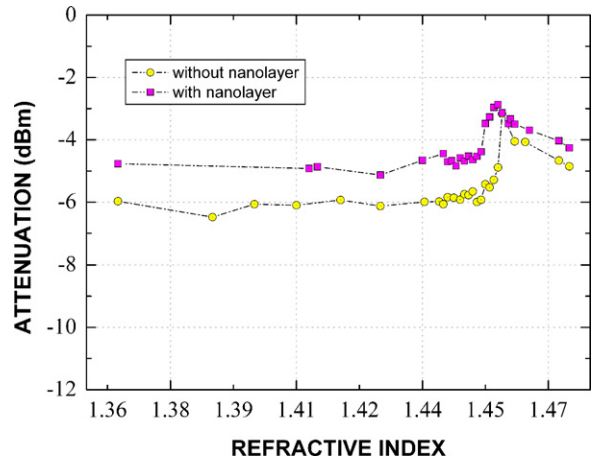


Fig. 9. Experimental values of the variation of the transmission of the LPG with the surrounding refractive index for LP<sub>14</sub>.

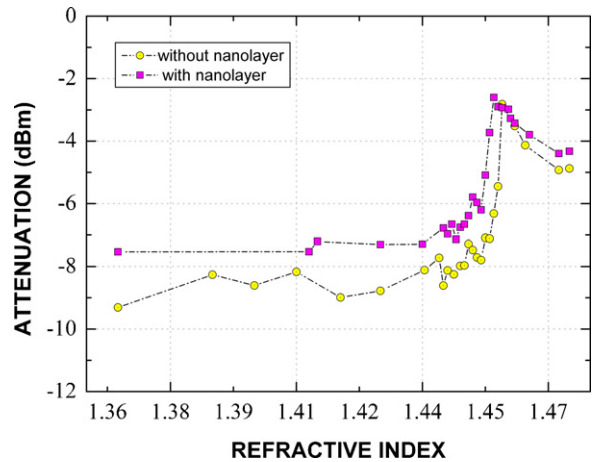


Fig. 10. Experimental values of the variation of the transmission of the LPG with the surrounding refractive index for LP<sub>15</sub>.

modes. However, it is observed that when the film is deposited there is a reduction in the attenuation bands when compared with LPG without film. This change is due to the fact that the refractive index of the thin film is higher than the cladding and this is able to favor the transition from cladding guided modes to overlay guided

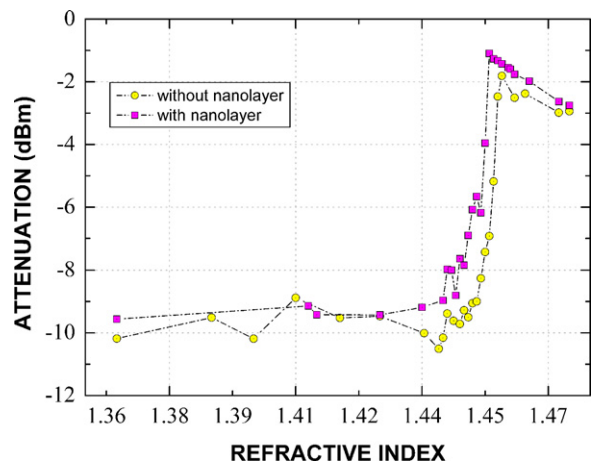


Fig. 11. Experimental values of the variation of the transmission of the LPG with the surrounding refractive index for LP<sub>16</sub>.

modes, causing a strong re-organization of the cladding modes, changing the wavelength center and the minimum transmission value of the attenuation band [13].

#### 4. Conclusions

The behavior of an LPG relatively to the variation of the refractive index of external media was studied in terms of wavelength shift and of transmission band variations. The most external attenuation band is the one that shows the highest sensitivity to the variations of transmission and wavelength with the surrounding refractive index. The results obtained demonstrated that the sensitivity in terms of wavelength is higher than in terms of transmission.

The experimental results demonstrate the possibility of tuning the sensitivity to refractive index higher than the cladding through deposition of a thin film. For instance, in the refractive index region around 1.46, values of  $\sim 2578 \text{ nm/riu}$  (Refractive Index Unity) for the LPGs with thin film were obtained. This corresponds to a sensitivity response improved by a factor of  $\sim 1.7$  when compared with the LPGs without thin film. The results obtained in this work have more experimental values than previously reported [10]. Ishaq et al. showed that an increase in sensitivity was due to the deposition of a thin film. However, the fiber used was B-Ge and it was not mentioned cladding order mode. In our case an SMF28 fiber was used which would result in an easier integration in communication systems indicating the feasibility to perform remote sensing. The results obtained demonstrate a possibility of measurement of parameters with refractive index higher than the cladding.

This optical fiber refractometer can be applied in different areas of study, such as fuel quality control and food chemistry, because it does not contaminate the samples (for example to control the degradation of food oils) and to quantify compounds in solution provided there is a change of the refractive index.

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**Eliana Simões** has a BS degree in biochemistry and food chemistry from the University of Aveiro, and a master degree in analytical chemistry and quality control from the University of Aveiro. Her principal research interests are fiber optic sensors and food chemistry.

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**Orlando Frazão** graduated in physics engineering (optoelectronics and electronics) from the University of Aveiro. He received his PhD from University of Porto, in 2009 on optical fiber sensors based on interferometry and non linear effect. He is currently working toward the PhD degree in physics at the University of Porto. Presently, he is a senior researcher at Optoelectronics and Electronic Systems Unit, INESC Porto.

**Paulo Caldas** graduated in applied physics (optics and lasers) from the University of Minho in 1999. He received the MSc degree in lasers and optoelectronics in the physics department of the University of Porto in 2003. Currently he is a researcher in the Optoelectronics and Electronic Systems Unit at INESC Porto working toward his PhD on optical fiber sensors based on evanescent field interaction in the physics department of the University of Porto.

**João de Lemos Pinto** has a PhD degree in Applied Physics from the University of Hull, England. Since 2000, he has the position of full professor of the Department of Physics of University of Aveiro, Portugal. He is presently leading the optics research group of I3N-Aveiro, Institute of Nanostructures, Nanomodelling and Nanofabrication, and the electronic and optoelectronic components research area of IT-Aveiro, Institute of Telecommunications. His present research interests include optical communications, Bragg gratings systems, optical image processing, holography and promotion of physics in society.