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High sensitivity humidity sensor based on cladding-etched optical fiber and lossy mode resonances

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a b s t r a c t

In this work a high sensitivity optical fiber humidity sensor (OFHS) is presented. The configuration chosen for this purpose is a cladding-etched single mode optical fiber (CE-SMF) coated with a thin film of tin oxide ($SnO₂$). The etching has been made using hydrofluoric acid (HF) and the coating has been fabricated by means of sputtering. Tin oxide was used to build the nano-coating which produces the Lossy Mode Resonance (LMR) and works as sensitive material. Theoretical and experimental results are shown and compared. The device was tested using a climatic chamber in order to obtain the response of the OFHS to relative humidity. Changes greater than 130 nm have been obtained for relative humidity varying from 20% to 90%, which gives a sensitivity of 1.9 nm/%RH.

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1. Introduction

Optical fiber humidity sensors (OFHS) offer several advantages over electronic humidity sensor like small size, lightness, the possibility of working on flammable environments and, the most important, their electromagnetic immunity. Other important advantage of OFHS over electronic humidity sensor is the possibility of working at higher temperature and pressure ranges.

It can be distinguished different ways of measuring relative humidity with an OFHS. The first way would be by amplitude based techniques which consist of measuring changes in the transmitted optical power caused by changes on the optical absorption or on the refractive index of the coating $[1-4]$. The other way is named wavelength based techniques, which overcome the limitations of the previous technique, such as external noises or power fluctuations [\[5\].](#page--1-0) Other techniques include measuring shifts on the phase of the light or measuring the state of polarization, but these methods require more complex setups.

For wavelength based techniques, transmission bands or absorption peaks in the transmission profile should be achieved in order to measure changes on the wavelength where that absorption peak is placed. Absorption peaks could be achieved using several structures, such as long period gratings (LPGs) [\[6–8\],](#page--1-0) pho-

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[http://dx.doi.org/10.1016/j.snb.2016.04.045](dx.doi.org/10.1016/j.snb.2016.04.045) 0925-4005/© 2016 Elsevier B.V. All rights reserved. tonic crystal fibers [\[9\]](#page--1-0) or interferometric structures [\[10–12\];](#page--1-0) and transmission bands could be achieved, for example, using a single mode-multimode-single mode structure [\[13\].](#page--1-0) Most of these structures generate an absorption peak at certain wavelength by creating a grating on the core of the optical fiber, modifying its refractive index or with interferometers. These structures present some desirable characteristics, such as small spectral width of the attenuation band. On the other hand, the dynamic range and sensitivity of these devices are generally small $[6-13]$. Another method for achieving an absorption peak is the generation of Lossy Mode Resonances (LMRs).

LMRs are electromagnetic resonances that are generated by some materials deposited over a waveguide. Then, coupling of light to the cladding modes happens at certain resonance wavelengths, without modifying the refractive index of the core, provided that the material meets the requirements for its generation. This condition is that the real part of the thin-film permittivity is positive and higher in magnitude than both its own imaginary part and the real part permittivity of the materials surrounding the thin-film [\[13\].](#page--1-0) There is a wide variety of materials that can be used to generate LMRs, e.g., polymers and semiconductors, allowing fabricating optical fiber sensor for multiple applications [\[13–20\].](#page--1-0) Generation of LMRs on single mode optical fiber (SMF) has generally been done with tapered optical fibers $[17,24]$ as the way to get access to the evanescent field. In this work a cladding-etched single mode optical fiber (CE-SMF) coated with a nano-coating has been used for the fabrication of the OFHS. Etching the optical fiber has some advantages over conventional tapered optical fibers, like providing a much more repetitive process from the point of view of the physical parameters of the resultant taper. The main difference between tapered and cladding etched optical fiber is that in the first case both the cladding and the core are narrowed, maintaining the relation core/cladding, whereas in the second case only the diameter of the cladding is reduced [\[23–25\].](#page--1-0) Development of adiabatic tapered optical fibered by means of chemical etching has been previously studied [\[24–27\]](#page--1-0) and some theoretical approaches can be found in [\[28\].](#page--1-0) There are also some OFHS that have employed chemical etching, for example, to improve the sensitivity of some interferometric devices [\[6\],](#page--1-0) or to taper an optical fiber and then, by adding an appropriate coating $[3,4]$, to develop an evanescent wave OFHS, generally based on changes on the optical absorption or the refractive index of the coating which leads to changes on the transmitted optical power. Chemical etching has been also applied to long period gratings optical fibers in order to improve their sensitivity [\[28\]](#page--1-0) to the external refractive index. Here, a nano-coating has been coated onto the CE-SMF in order to achieve the LMR which allows to measure changes in the wavelength where the LMR occurs.

The material that has been chosen for the fabrication of the nano-coating is tin oxide $(SnO₂)$. The etched optical fiber was coated with this material by means of sputtering because it is a method that provides homogeneous films with good control of the thickness deposited. Tin oxide has a relatively high refractive index; it is sensitive to changes on the relative humidity and besides, it meets the requirements for generating LMRs. Several materials have been previously studied as LMR generators and as humidity sensors, most of them coating a plastic cladding removed optical fiber (200 µm), such as PAH/PAA [\[16,29\],](#page--1-0) ITO $[15,30]$, In_2O_3 $[28,29]$, TiO_2/PSS $[17,21]$ and SnO_2 $[31]$ etc. obtaining the best sensitivity for [\[21\],](#page--1-0) with values of 0.4 nm/%RH, 0.833 nm/%RH, 0.133 nm/%RH, 0.935 nm/%RH, 1.43 nm/%RH and 0.107 nm/RH respectively. Devices developed in previous works have been fabricated by dip-coating and a post-annealing process or by Layer-by-Layer assembly, which are slow methods and they also present some dependence on the environmental conditions. Furthermore, polymeric coatings [\[8,10\]](#page--1-0) present greater hysteresis and higher response times than the device developed here.

The device developed in this work improves the sensitivity of previous devices [6,7,17,20-22,30-32], it is relatively simple to fabricate and it has a smaller FWHM than other devices based on LMRs [\[17,31\].](#page--1-0) Besides it has short response times, good linearity and low hysteresis. To our knowledge, this is the first time that a claddingetched single mode fiber is used for the fabrication of a Lossy Mode Resonance sensor. The manuscriptis organized as follows: firstly, in Section 2, the structure, the fabrication of the device and the experimental set-up are analyzed. In Section 3 the simulated results, obtained with FIMMWAVE ® software, are shown and explained. In Section [4](#page--1-0) the device performance is discussed, and finally some concluding remarks are exposed in Section [5.](#page--1-0)

2. Device fabrication

2.1. Chemical etching

For the chemical etching, hydrofluoric acid (HF) diluted at 40% (Sigma-Aldrich) was used. Then the SMF (9/125 μ m, core/cladding diameter) purchased from Telnet RI, with 5 mm of the buffer removed, was immersed in the HF. When the external diameter reaches 60 μ m, the etched SMF was washed with water to remove all the HF. Next step is to immerse the SMF in 20% diluted HF to have fine control on the etching process until the external diameter reaches 19 \upmu m. This diameter has been proved to be good enough for getting access to the evanescent field and obtaining the LMR. Finally it was washed again with water to remove the HF and it was attached to a U-holder to keep the fiber straight for next steps. The cladding-etched optical fiber can be seen in [Fig.](#page--1-0) 1.

2.2. Sputtering coating

The CE-SMF was introduced in the vacuum chamber of the sputtering machine, which was a Pulsed DC-Sputtering System (Nadetech Inc.) and coated with tin oxide $(SnO₂)$. The $SnO₂$ target, 99.99% of purity, was purchased from ZhongNuo Advanced Material Technology Co. The sputtering process was done at 9×10^{-3} mbar and 180 mA and when half of the time has been completed (6 min), it has been turned 180◦ and coated for other 6 min. The thickness of the coating has been estimated by means of an AFM (Veeco Innova model 840-012-711) to be 140 nm for device D1. This thickness causes a LMR located at 1550 nm. If the thickness of the $SnO₂$ decreases the LMR shifts to lower wavelengths due to its sensitiveness to the surrounding medium refractive index [\[5,19\].](#page--1-0) On the other hand, if the thickness increases the LMR shifts to greater wavelengths until disappear, and other LMR will appear. Second LMR is less sensitive than the first, and so on. That will be demonstrated by developing other devices, D2 to D4, coated by a DC Sputtering System (Quorum K750X) at 9×10^{-2} mbar and 90 mA. Thickness of device D2 is 275 nm, thickness of device D3 is 250 nm, while D4 has a coating thickness of 650 nm, which means that the third LMR has appeared. In [Fig.](#page--1-0) 2 it is shown the refractive index of the coating which has been measured by ellipsometry (Uvisel 2, Horiba). For the wavelengths where the device works the coating has a refractive index of 1.92 and a low extinction coefficient.

2.3. Experimental set-up

When the sensor was fabricated, the optical fiber was spliced to SMF pigtails connected to the light source and the optical spectrum analyzer (OSA). The light source HP-83437A was a superluminescent emitting diodes (SLED) white light source which has four LEDs at 1200, 1310, 1430 and 1550 nm and the OSA was the HP-86142A. The optical fiber was introduced in a climatic chamber (Angelantoni ACS CH 250), where the relative humidity has been increased from 20% to 90% at a constant temperature of 25 \degree C. The experimental set-up is shown in [Fig.](#page--1-0) 3.

3. Simulations

In this section the results obtained by simulations with FIMMWAVE® software are shown. The effect of the water adsorbed onto the tin oxide layer can be modelled by the following phenomena. Since water is a polar molecule, the negatively charged oxygen of the water molecule is electrostatically attracted to the positively charged cationic side of the metal oxide surface. If the charge density of the cationic side is low, then water remains physically adsorbed at the surface by a weak electrostatic field $[32]$. When the cationic charge density is high, the electrostatic force is high enough to form a chemical bond between hydrogen and oxygen of a water molecule, which in turn may break the bond between oxygen and one of the hydrogen atoms [\[33–35\].](#page--1-0) Mostly, the force is high enough to break the bond in the initially adsorbed water vapour layer. Therefore, the initial monolayer is generally chemisorbed [\[32\].](#page--1-0) As relative humidity (RH) increases, an additional layer of water molecules starts to be formed, on the chemisorbed one. Many more physisorbed layers will be joined as humidity gets higher [\[36\].](#page--1-0) These layers are easily removed by decreasing the humidity [\[37,38\].](#page--1-0)

Different parameters have been taken into account for these simulations. On one hand, we have simulated how the device would behave for different optical fiber diameters with the same thickness of tin oxide. Diameter of the optical fiber has been swept from Download English Version:

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