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Nanostructured Materials in Optical Fiber Sensing

M. Hernaez*, C. R. Zamarreño, J. Goicoechea, I. R. Matias and F. J. Arregui

Electrical and Electronic Engineering Department, Universidad Pública de Navarra: Edif. Los Tejos, Campus Arrosadia, 31006 Pamplona, Spain

Abstract: This work comprehends a review of nanostructured materials employed in the fabrication of optical fiber sensors in the last years. The continuous advances in nanofabrication techniques have enabled to manipulate the matter precisely producing well defined nanostructurated coatings or repetitive patterns at nanoscale level. The interactions of light with these nano-organized materials or patterns at the nanoscale level enable to observe interesting phenomena, such as interferometry, fluorescence, absorbance, resonances and many others which can be exploited in the fabrication of sensing devices. A particular case consists of optical fiber sensors, where the light travelling through an optical fiber interacts with the sensitive layer. The properties of the sensitive layer, such as the organization, physical properties, chemical bounds etc. will determine the sensing characteristics of the final device. The utilization of some of the most common nanostructured materials, such as polymers, nanoparticles, metals, metal oxides or biological coatings are reviewed here.

Keywords: Optical fiber sensors, thin-films, nanostructured coatings, refractometers, biosensors, chemical sensors, metals, metal oxides, polymeric coatings.

1. INTRODUCTION

Optical fiber sensors have been established as an emerging technology in many different fields such as biomedicine, environmental control, food quality test or navigation systems [1]. These sensors rely on different physical or chemical principles and can measure multiple magnitudes, for example pressure, temperature or chemical compounds concentrations [2-4]. The very well-known advantages of optical fiber sensors comprise, among many others, the immunity to electromagnetic interferences, easy multiplexation, low weight and transmission losses, small size or real time monitoring [5,6].

Much research has been done in the field of thin-film coated optical waveguides within the last decades [7,8]. Nanostructured thin-films when used in conjunction with optical fibers can dramatically improve the performance and functionality of optical fiber sensors [9-12]. However, the fabrication of these micro and nano-coatings is not trivial and requires in most cases a multidisciplinary knowledge [13].

Different thin-film coated optical waveguide configurations have been studied in order to exploit the advantages of the optical fiber configuration in the fabrication of sensing devices, and many of them have been described in different sensing applications and patents [14].

In the next sections, it will be described the utilization of different optical fiber configurations combined with diverse nanostructured materials for the fabrication of optical fiber sensors based on different sensing principles such as interferometry, fluorescence, absorbance or electromagnetic resonances.

*Address correspondence to this author at the Electrical and Electronic Engineering Department, Universidad Pública de Navarra: Edif. Los Tejos, Campus Arrosadia, 31006 Pamplona, Spain; Tel: +34948166044; Fax: +34948169720; Email: miguel.hernaez@unavarra.es

2. INTERFEROMETRIC NANO-CAVITIES

One of the simplest structures that can be fabricated using nanostructured coatings onto optical fibers is an interferometer. It consists of a nanocoating deposited onto the perpendicularly cleaved end of an optical fiber. This coating forms an interferometric cavity schematically represented in (Fig. **1**) [15].

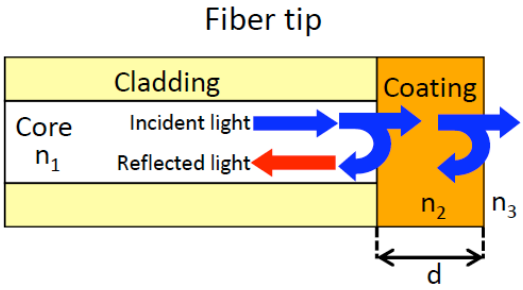

Fig. (1). Schematic representation of an interferometric cavity at the end of an optical fiber.

In this interferometric cavity, two optical mirrors are formed due to the different refractive indices of the optical fiber core (n1), the deposited coating (n2) and the surrounding medium (n3).

The reflectivity depends on the thickness of the coating and the refractive index of the three materials involved (optical fiber core, coating and external medium). Assuming that the optical fiber refractive index is fixed, the reflected power will change if there are changes in the refractive index of the external medium or if the coating suffers any variation. Therefore, in order to detect changes in any magnitude, it is

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just necessary to use a material sensitive to this magnitude to create the coating. This way, the variation in this parameter will produce a variation in the reflected optical power that can be measured with a typical reflection experimental setup.

Light launched from a light source pass through an optical coupler and reaches the interferometer. Part of the optical power is transmitted to the external medium, and the rest is reflected. This reflected power can be measured in the detector after passing again through the coupler. As the thickness of the cavity is shorter than the coherence length of a LED source, it is possible to use this kind of sources, avoiding the use of lasers [16-20]. This fact and the simplicity of this architecture have allowed the development of many kinds of optical fiber sensors based on it.

Different humidity sensors based on interferometric cavities have been developed in the last years. As it has been stated above, a change in the effective refractive index of the deposited coating will produce a variation in the reflected power. This way, if this coating is made of a material which thickness or refractive index is sensitive to humidity changes, the reflected power will represent these variations in the relative humidity of the surrounding medium. For example, in [21], an optical fiber humidity sensor based on SiO_2 nanoparticles is presented. It shows a good sensitivity and really fast rise and fall times, improving previous humidity sensors that used polymeric coatings [22].

The utilization of films that change their thickness when the pH of the external medium varies (swelling/deswelling effect), such as hydrogels or polymers [23], has enabled the fabrication of repetitive and robust pH sensors. In [24] the well known poly(allylamine hydrochloride) / poly(acrylic acid) (PAH/PAA) structure [25-28] is deposited onto an optical fiber tip to create a pH-sensitive nanocavity.

Interferometric cavities have been used to detect different volatile organic compounds (VOCs). For example, Consales *et al.* developed NO_2 detectors based on this architecture [29-31]. Moreover, ethanol and other VOCs detectors based on vapocromic compounds nanocavities have been fabricated [32-34].

In the last years, some biosensors based on optical fiber interferometers have been developed. For example, in [35,36] a hollow core fiber fragment is used to create a gap between a SMF and the sensing film made of chitosan and PSS. This way, a bovine serum albumine sensor has been fabricated.

3. FLUORESCENT SENSORS

An important group of optical fiber sensors consist of those based on fluorescence or phosphorescence measurements, referred as fluorescence henceforward. Fluorescence-based optical fiber sensors have been used traditionally in applications such as analytical chemistry, biochemistry, photochemistry, cellular biology, medical diagnosis or biotechnology for the detection of different compounds with high sensitivity and specificity [37-41]. The fluorescent or phosphorescent dye, used as transducer can be adhered to the optical fiber and is usually entrapped in a matrix with high permeability to the measurand [42-44]. Thus, the sensitivity of the optical sensor depends on both the fluorescence intensity of the dye and the matrix characteristics, such as its density, viscosity, hydrophobicity, transparency etc. [45].

Concerning the fluorescence intensity (I_f), it is in general proportional to the excitation intensity (I_e) and the dye concentration ([D]), which also depends on the quenching effects. It is also important to take into account the fluorescence efficiency (η) of the dye, in other words, the ratio between the photons absorbed by the material and the photons emitted by fluorescence or phosphorescence mechanisms. This relation is expressed by Parker's law in 1.

$$I_f = 2.3 \cdot K \cdot l\,\varepsilon \cdot I_e \cdot \eta \cdot [D] \quad (1)$$

where l is the path length of the light within the detection layer, ε is the molar absorption coefficient and K is a fitting factor associated to the geometry of the measurement instrument. However, most of the detection systems do not quantify directly the analyte concentration from the fluorescence intensity but using and indirect approach that compares the fluorescence intensity in absence of analyte (I_o) and the fluorescence intensity in the presence of the analyte (I_q) which is given by the Stern-Volmer equation in 2 [46].

$$I_o/I_Q = 1 + K_{SV}[Q] \quad (2)$$

where [Q] is the quencher concentration and K_{sv} is the Stern-Volmer constant, given by the expression $K_{sv}=\tau_o k_q$, where k_q is the bimolecular deactivation speed constant and τ_o is the excitation state time.

As it was advanced before, fluorescence emission can be expressed as a function of the quencher target molecule concentration but it is also necessary to take into account other effects that depend on the fluorescent material itself such as the photodegradation or photobleaching that occurs when the indicator is exposed to the excitation source for a long time [47], the selfquenching or self-absorption at high concentrations of fluorophore and the leaking or diffusion loses of the fluorescent indicator molecules through the supporting matrix. In general, the design of an optimum fluorescent sensor requires the selection of dyes with long unquenched state lifetime and matrices with high permeability to the measurand, good mechanical and chemical stability, and none or low interference with the measurements [48, 49].

Fluorescent molecules have been traditionally used in the fabrication of optical fiber sensors [45]. These molecules can be easily entrapped into a supporting matrix and adhered to the optical fiber using different fabrication techniques and optical fiber configurations as it is summarised in (Table **1**). For instance, the fluorescent dye 1-hydroxy-3,6,8-pyrene trisulfonic acid trisodium salt (also known as pyranine or HPTS) has been used in pH sensing applications [50] as well as CO_2 detection in gaseous [51], aqueous [52] or blood [53] media. In the same manner, ruthenium-based coatings have been applied in the fabrication of optical fiber sensors for pH [54, 55] and O_2 [56, 57] detection, fluorescein-based compounds have been also applied to obtain optical fiber sensing probes for pH [58-60] or cocaine [61] and eosin red fluorescent dye has been used for pH [62] and ammonia [63] detection by means of different optical fiber configurations. Fluorescent polymers have been also used for the detection of explosives [64], Cu^{2+} [65] or Na^{+} [66] ions employing thin-films fabricated onto fiber end tips, decladded MMFs or MOFs respectively.

Table 1. Summary of Optical Fiber Sensors Based on Fluorescent Dyes

Fluorescent Indicator	Target	Supporting Matrix	Fabrication Technique	Optical Structure	Reference
HPTS	pH	propyltriethoxysilane and (3-glycidoxy)propyl trimethoxysilane	Sol-gel	V-tapered fibers	[50]
		polyelectrolytes	LbL		[67]
				MMF end tips	[47]
	Gaseous CO_2	n-octyltriethoxysilane (Octyl-triEOS)/tetraethylorthosilane (TEOS)	Sol-gel dip-coating		[51]
	Aqueous CO_2	Hybrid xerogels			[52]
	pCO_2 in blood	Hydrophobic sol-gel glass	Sol-gel		[53]
Ruthenium complexes	Aqueous O_2	TEOS/MTEOS	Sol-gel	U-bent POF	[57]
				Spiral-shaped POF	[56]
	pH	tetraethoxysilan and phenyltriethoxysilan		Fiber End tips	[54]
		polyelectrolytes	LbL		[55]
Fluorescein complexes		Hydrogel-matrix	Sol-gel	Side-polished fiber	[58]
		tetraexthoxysilane (TEOS)	Sol-gel	V-tapered fibers	[59]; [60]
	cocaine	Molecularly imprinted polymer	Polymerization	MMF end tips	[61]
Eosin	pH	cellulose acetate (CA)	Liquid flow	MPOF	[62]
	ammonia			MOF	[63]

Other materials, such as functionalized carbon dots and vapochromic compounds can also exhibit fluorescence when excited at certain wavelengths. The LbL technique has been used successfully to immobilize both carbon dots and vapochromic compounds onto optical fiber end tips for the detection of Hg^{2+} ions [68,69] and VOCs [32] respectively. Similarly glucose/galactose binding proteins functionalized with a fluorescent dye have been adhered to an optical fiber end tip within a hydrogel matrix and used for continuous glucose monitoring in animals [70]. Here, the conformational change of the protein in presence of glucose inhibits the fluorescence as it is shown in (Fig. **2**).

In this context, luminescent semiconductor nanocrystals, or quantum dots (QDs), are particularly attractive. The unique optical properties of QDs, such as the particle-size dependent luminescence, high efficiency, narrow fluorescence emission band, broad absorption spectrum and high photostability when compared to those of traditional molecular fluorophores, can provide new solutions to many of the problems associated with traditional luminescence sensors and are the promise for a completely new set of applications using different optical fiber configuration [71]. As an example, CdTe quantum dots of various sizes have been embedded in polymeric films using the Layer-by-Layer technique in order to obtain temperature sensors by means of different optical fiber configuration schemes. In [72], the structures have been fabricated onto MMF tapered ends while in [73] and [74] the structures have been fabricated in the inner part of HCFs and in the holes of MOFs respectively (see Fig. **3**), which protects the QD nanofilm from the environment while it is still sensitive to temperature changes reducing considerably the effects of photobleaching. Moreover, the utilization of quantum dots of various sizes enabled the utilization of several reference signals [73]. Optical fiber sensing probes for Cu^{2+} detection were also fabricated in [75] by immobilizing CdSe/ZnS quantum dots onto optical fiber tapered end tips using the sol-gel dip-coating technique.

4. ABSORBANCE-BASED SENSORS

The transmission of the light through an analyte (T) follows Lambert-Beer's law (ec. 3)

$$T = I/Io = 10^{-\alpha l} = 10^{\varepsilon [C] l} \quad (3)$$

where I_o and I_f represent the light intensity before and after passing through the sensitive region respectively, l is the path length of the light within the absorbing material and α is the absorption coefficient of the indicator, which can be expressed by the product between the molar absorption coefficient (ε) and the concentration [C] of the target. The

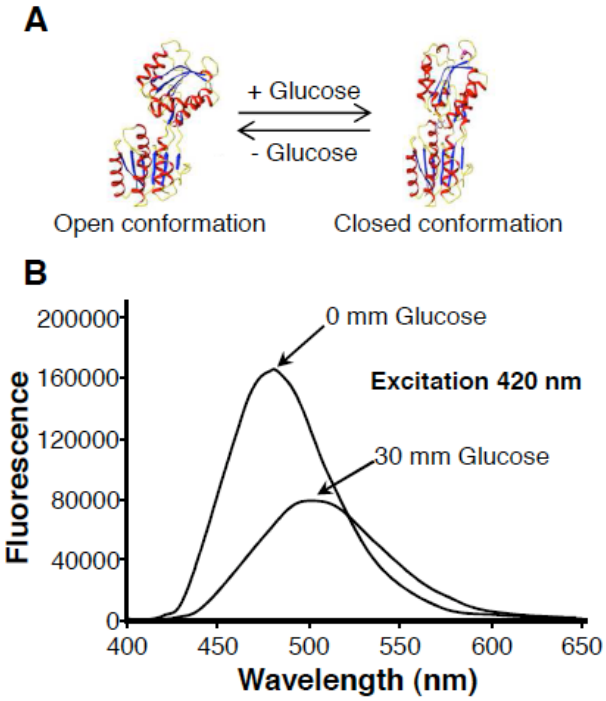

Fig. (2). (A) Ribbon structures depicting the glucose-mediated conformational change of glucose/galactose binding protein (GGBP). Left: Open unliganded conformation of GGBP usin Protein Data Bank structure file 2FW0. Right: the closed or glucose-bound structure of GGBP using Protein Data Bank structure file 2FVY. (B) Fluorescence response of acrylodan-labeled GGBP to glucose. The shaded regions show wavelength bands monitored by the optical system. Extracted from [70] with permission from Elsevier.

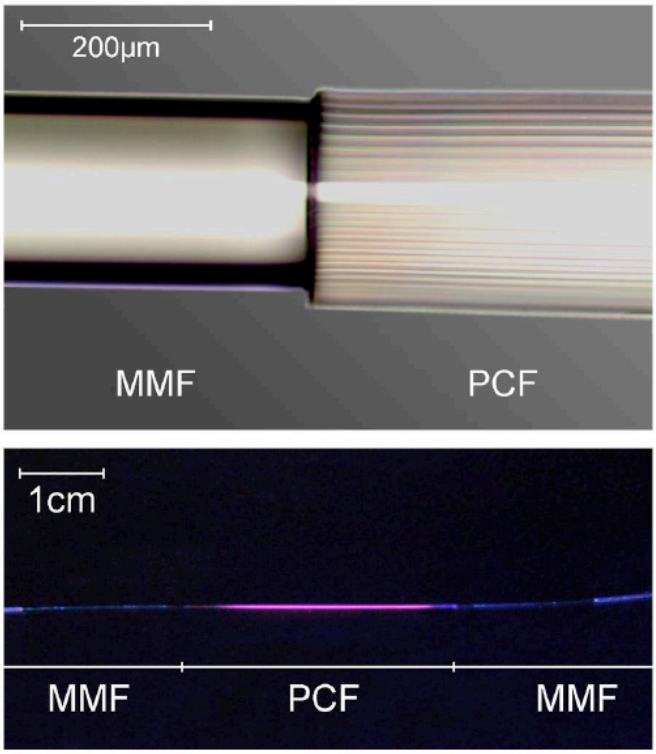

Fig. (3). Top: Microscope image of the MOF-MMF splices. Bottom: Picture of the optical fiber arrangement under UV illumination. In the middle region the red quantum dots' fluorescence from the MOF inner holes can be seen. Extracted from [74] under Creative Commons License.

logarithmic relation of ec. 3 can be also expressed in terms of absorbance as a linear function of the absorbing material concentration (eq. 4).

$$A = -\log_{10} I/Io = \alpha l = \varepsilon [C] l \quad (4)$$

Absorbance-based optical fiber sensors are often used in optical fiber sensing owing to the readout simplicity associated to these devices. The utilization of these sensors relies on the characteristic light absorption of every material and its spectral signature. However, this requires complex discrimination algorithms and sometimes tedious learning process in order to obtain optimum results [76-78]. An alternative approach comprises the utilization of an active indicator as the transducer. The generic idea is that the active indicator changes its optical properties (absorbance) depending on the presence or concentration of the sole target to be measured, which enables to determine the target concentration. In order to maximize the interactions of the light with the indicator, the latter can be adhered, directly or immersed in a supporting matrix, to the fiber surface. Therefore, the matrix properties together with the optical fiber geometry will determine the penetration depth of the evanescent field within the sensitive structure and it will be crucial in the overall response of this kind of sensors [79]. The next paragraphs will focus the attention in the utilization of thin absorbing films adhered to different optical fiber schemes for the fabrication of optical fiber sensors as it is summarized in (Table **2**).

In general, the optical fiber schemes employed are intended to grant the major access to the light travelling through the optical fiber core in order to maximize the interactions of the light with the active thin-film. Bent fibers can be an interesting choice to increase the evanescent field and hence the interactions with the surrounding active media as it is described by several authors for the fabrication of humidity [80,81], pH [82], ammonia [83], H_2O_2 [84] and Hg^{2+} [85] sensors.

An alternative approach and one of the preferred embodiments due to its simplicity consists of the removal of the optical fiber cladding and the fabrication of the thin-film directly onto the optical fiber core. This, configuration is usually referred as cladding removed (CR) followed by the optical fiber type such as multi-mode fiber (MMF) or plastic optical fiber (POF) and have been largely exploited in literature for the development of optical fiber sensors for ammonia [86,87], humidity [88,89], toluene [90], H_2S [91,92], aerosol [93] or pH [94-104] detection.

Other options aimed to maximize light interactions with the sensitive film comprise the modification of the regular optical fiber structure or the utilization of special optical fibers. As regard as the modification of the optical fiber structure it can be mentioned the fabrication of optical fiber sensors by means of hetero-core optical fibers (HCOF) [105-107], tapers [22, 108, 109] or side-polished fibers

Table 2. Summary of Optical Fiber Sensors Based on Absorbance Measurements

Optical Fiber Configuration	Target	Active Coating	Fabrication Technique	Reference
CRMMF/CRPOF	ammonia	SiO_2/PDDA	LbL	[86]
		Tetrakis-(4-sulfophenyl)porphine(TSPP)		[87]
	Humidity	Polymer fibers	Electrospinning	[88]
		agarose	sol-gel	[89]
	Toluene	TiO_2/SiO_2		[90]
	H_2S	Ag	Sputtering	[91,92]
	Aerosol	Thymol blue + TEOS	sol-gel	[93]
	pH			[94]
		cresol red, chlorophenol red, bromophenol blue + CNTs		[115]
		Cresol red, chlorophenol red, bromophenol blue		[95,96]
		Ethyl violet		[97]
		Bromophenol blue		[98]
		Bromocresol green & cresol red		[99]
		silica matrix		[100]
		methyl orange		[101]
		polymeric film	LbL	[102]
		neutral red		[103]
Bent fibers	pH			[82]
	Hg^{2+}	PVC/bis(2-ethylhexylsebacate)		[85]
	Ammonia	Bromocresol purple/SiO_2	sol-gel	[83]
	H_2O_2	Ti(IV)-oxyacetylacetonate doped Nafion		[84]
	Humidity	Agarose		[80]
		SiO_2/methylene blue		[81]
Tapered fibers		PDDA/poly-R	LbL	[22,80,108]
	Alcohol	Novolac resin and PVDF	sol-gel	[109]
Side polished fiber	pH	Cresol red, chlorophenol red and bromophenol blue		[110]
	H_2	Pd/WO_3	sputtering	[111]
Hetero-core fibers		Pd/Au	evaporation	[105,106]
	Humidity	poly (ethylene oxide)	sol-gel	[116]
HCFs	pH	Phenol red & cresol red		[112]
	Humidity	Polymer	LbL	[108]
		Poly-glutamic acid/poly-lysine		[107]
MOFs	H_2	Pd	Thermal evaporation	[113]
	HCl	Porphyrin-doped TiO_2	sol-gel	[114]

[110, 111] while the use of special optical fibers for the fabrication of sensors is mainly referred to hollow-core fibers (HCFs) [112, 108] and microstructured optical fibers (MOFs) [113, 114].

5. RESONANCE-BASED SENSORS

When an optical waveguide is coated by a thin-film (see Fig. **4**), the propagation of light is affected. Depending on the properties of the different materials involved in the system (the waveguide, the coating and the external medium), three different cases of electromagnetic resonances can be distinguished [117].

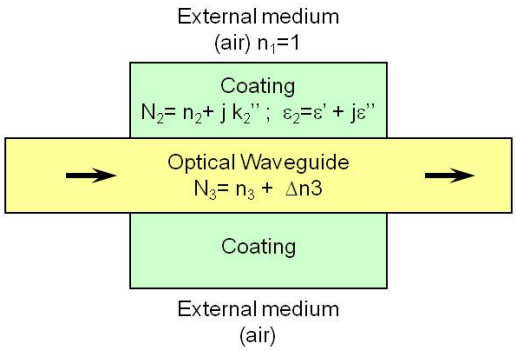

Fig. (4). Schematic representation of the optical system used to obtain electromagnetic resonances.

The first case occurs when the real part of the thin-film permittivity is negative and higher in magnitude than both its own imaginary part and the permittivity of the material surrounding the thin film. In this case, a resonance called Surface Plasmon Resonance (SPR) is produced.

The second case occurs when the real part of the thin-film permittivity is positive and higher in magnitude than both its own imaginary part and the permittivity of the material surrounding the thin film. In these conditions a second type of resonances called Lossy Mode Resonance (LMR) is produced.

Finally, the third case occurs when the real part of the thin-film permittivity is close to zero, while the magnitude of its imaginary part is large. This case, known as long-range surface exciton polariton (LRSEP), has not been applied to the fabrication of optical fiber sensors and will not be included in this review.

The system showed in (Fig. **4**) can be easily adapted to optical fiber. The uncladded core of an optical fiber is used as a waveguide and the appropriate coating is deposited onto it. This device is connected in both extremes to an optical source and a detector to obtain the complete interrogation setup [118].

When an electromagnetic resonance (EMR) (SPR or LMR) is produced, the generated absorption peak shifts to different wavelengths when the refractive index of the external medium changes.

SPR have become an optical fiber sensor standard in the last years, with a lot of research done in this field. For example, different optical fiber refractometers based on SPR have been developed by using MMF, SMF or tapered fiber-schemes [118-125].

If the SPR supporting thin film (usually a metal) is coated with any material which refractive index is sensitive to some magnitude, a variation in this magnitude will produce a shift in the SPR absorption peak. This architecture has been applied for the fabrication of different optical fiber sensors. For example, in [126] a pH sensor was fabricated by adding a pH-sensitive hydrogel to a SPR supporting coating. In addition, different sensors based on SPR have been developed for the detection of volatile organic compounds (VOC), such as alkanes [127, 128] or ammonia [129].

However, the fabrication of biological sensors is the field where SPR-based optical fiber sensors have acquired the main interest [130, 131]. These sensors include biologically active structures that detect the target, such as bacterial cells, enzymes, antibodies and other proteins and allow the detection of proteins [132], viruses [133, 134], pesticides [135], living cells [136], etc.

As it has been mentioned, the apparition of SPR devices has supposed an important breakthrough in the field of optical fiber sensors. But this phenomenon have some limitations, such as the necessity of using polarized light or the fact that it is only generated by some specific metals.

Recently, the first optical fiber sensors based on LMR have appeared using LMR supporting coatings from different nature. Thus, optical fiber refractometers including different metal oxides have been fabricated [137-139]. These sensors present sensitivities in the range of similar SPR-based sensors, but the experimental setup and the fabrication procedure are simpler.

In addition, sensors based on these refractometers have been developed by just adding a sensitive coating to the previous device. For example, polymeric coatings were deposited onto the LMR supporting devices in order to obtain humidity and pH sensors [27, 140, 141].

However, metal oxides are not the only materials that can generate LMR. In [142] a refractometer based on TiO_2 nanoparticles is presented and in [143], a polymeric coating is simultaneously used as LMR supporting coating and sensitive coating in order to obtain a pH sensor with a really fast and sensitive response and low hysteresis (Fig. **5**).

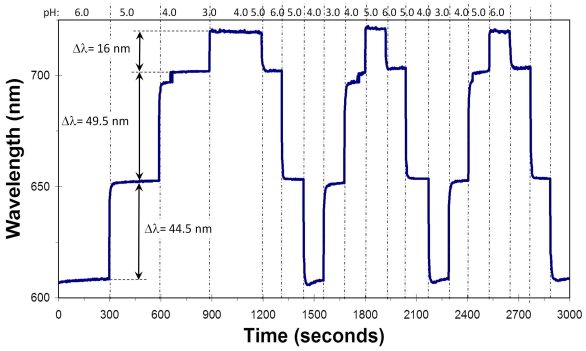

Fig. (5). Dynamical response of a pH sensor based on LMR generated onto a polymeric coating. Extracted from [143] with permission from Elsevier.

Table 3. Summary of Optical Fiber Sensors Based on Electromagnetic Resonances

Optical Fiber Configuration	Target	Active coating	Fabrication Technique	Reference
SPR	Refractive index	Au, Ge, Ag, SiO2	RF sputtering	[144]
		Au	Evaporation	[119]
		Au	Layer by Layer	[123]
		Au		[118]
		Ag, ZrO	Sol-Gel	[124]
		Al	RF sputtering	[125]
	Alkanes	Ag/Pt	RF sputtering	[127]
	Hydrogen	Au/Pd		[106]
	pH	Smart hydrogel	Silanization	[126]
	Ammonia	Polyaniline	Sol-Gel	[129]
	Living cells reactions	Au	RF sputtering	[136]
	pesticides	acetylcholine esterase	Gel entrapment	[135]
	DNA hybridization	DNA aptamer bioreceptors	Chemical adsorption	[145]
	Cadmium	phytochelatins		[146]
	SARS coronavirus	anti-N-1 monoclonal antibody		[134]
	Influenza A virus	Antibodies against the hemagglutinin		[133]
	alpha-fetoprotein	Antigen – antibody sandwich		[132]
	thrombin	Human thrombin binding aptamer		[147]
LMR	Refractive index	ITO	Sol-gel	[137,138]
		InO		[139]
		TiO2/PSS	Layer by layer	[142]
	Relative Humidity	ITO + Agarose	Sol gel	[140]
		ITO + PAH/PAA	Sol gel + LbL	[27]
	pH	PAH/PAA	Layer by Layer	[141,143]
	VOCs	ITO	Sol gel	[148]

In (Table **3**) a summary with different examples of optical fiber sensors based on SPR and LMR is shown.

6. CONCLUSIONS

This paper has reviewed four categories of nanostructured optical fiber sensors based on fluorescence, absorbance, interferometric nano-cavities and electromagnetic resonances. All these sensors rely on the interactions of light with the nanostructured thin-films. The sensing mechanism behind each category and current state of the art has been presented.

7. FUTURE DEVELOPMENTS

In the last few years, different studies have shown the excellent potential of carbon derivates as sensitive materials for the detection of ambient pollutants. The particular structure of these carbon-based materials and their unique properties such as their tensile strength, high surface area, low density as well as their exceptional electrical and thermal properties make them very attractive to produce small and portable sensors usable on many different substrates [149] such as optical fibers [30, 29, 150]. A step forward could be the functionalisation of these carbon-based structures that can

include fullerenes, graphene films, carbon nanotubes, nanoporous carbon films and diamond like carbon films among others in order to increase their sensitivity and minimize the undesired effects like cross-sensitivity [151].

Additionally, the utilization of metal-organic framework materials could also expand the challenges in the design and synthesis of structures with exceptionally high surface areas. (up to 3,000 $m^2 \cdot g^{-1}$) [152] while the investigation of the sensing properties of these porous materials combined with optical fibers is still an unexplored field.

CONFLICT OF INTEREST

The authors confirm that this article content has no conflicts of interest.

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