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Tailoring properties of lossy-mode resonance optical fiber sensors with atomic layer deposition technique [☆]

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ABSTRACT

The paper shows application of atomic layer deposition (ALD) technique as a tool for tailoring sensorial properties of lossy-mode-resonance (LMR)-based optical fiber sensors. Hafnium dioxide (HfO_2), zirconium dioxide (ZrO_2), and tantalum oxide (Ta_xO_y), as high-refractive-index dielectrics that are particularly convenient for LMR-sensor fabrication, were deposited by low-temperature (100°C) ALD ensuring safe conditions for thermally vulnerable fibers. Applicability of HfO_2 and ZrO_2 overlays, deposited with ALD-related atomic level thickness accuracy for fabrication of LMR-sensors with controlled sensorial properties was presented. Additionally, for the first time according to our best knowledge, the double-layer overlay composed of two different materials - silicon nitride (Si_xN_y) and Ta_xO_y - is presented for the LMR fiber sensors. The thin films of such overlay were deposited by two different techniques - PECVD (the Si_xN_y) and ALD (the Ta_xO_y). Such approach ensures fast overlay fabrication and at the same time facility for resonant wavelength tuning, yielding devices with satisfactory sensorial properties.

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1. Introduction

Nowadays optical sensors already allow for measuring majority of physical and chemical measurands of interest [1]. These are known various temperature [2], [3], pressure [4,5], strain [3], displacement [6], electromagnetic field [7–9] and refractive index [10,11] sensors, as well as chemical [12] and biosensors targeted towards certain molecule or even whole biological cell [13–15] to name only a few. While the modern optical sensors technologies cover a wide range of device architectures, physical effects and sensing strategies, the sensors often require deposition of thin films (overlays) or surface structures. The properties of these films or structures can be various and in particular they can be metallic or dielectric. The mentioned overlays or structures are used to initiate or modify sensorial response of the sensors, allowing for precise adjustment of the sensors properties according to the needs. Therefore in most cases the thin overlay thickness and its optical properties (primarily the complex refractive index - n^*) should be strictly controlled.

Contemporary thin film fabrication techniques are expected to provide high deposition accuracy and process-to-process reproducibility, as well as coating uniformity on various substrates and

shapes. These circumstances impose stringent requirements on control of the overlay deposition rate and the overlay properties on different stages of deposition. The matter of technological requirements becomes even more critical when three-dimensional objects like optical-fiber-based sensors are considered. All these make the matter of choice of the thin film deposition technique crucial.

Many nanofilm deposition techniques have already been used in the field of optical sensing, such as Langmuir-Blodgett method [16], spin coating [17], sol-gel process [18], layer-by-layer technique [19], chemical vapor deposition (CVD) [20], or physical vapor deposition (PVD) methods, which include evaporation [21] or sputtering [22]. They all have specific drawbacks, such as poor deposition process control, low coating robustness or lack of accuracy and coating uniformity.

Atomic layer deposition (ALD), which is a specific variation of the CVD method, seems to be the first-choice technique enabling control of the sensors properties by means of precise adjustment of the overlay features. The ALD chemical precursors are delivered to the reaction zone separately in time, and hence the complementary and sequentially repeated chemisorption half-cycle reactions responsible for the layer growth undergo at a surface in a self-limiting manner [23]. Thanks to this very special paradigm an atomic control of the layer thickness is possible, with uniquely conformal, uniform and tight layers, even when they are deposited

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on complicated high-aspect-ratio surfaces. These and other advantages of ALD, such as capability for deposition of dense layers in a wide range of materials (oxides [24,25], nitrides [26–28], metals [29,30] and many others isolators, semiconductors and conductors), possible deposition at relatively low temperatures (even a way below 100 °C [31,25]) lead to a situation where the ALD technique is a must in nowadays microelectronics [32,33]. At the same time, it reaches more and more applications in other very diverse branches [34,35], and has already begun to be used in optical sensors technology too [36–42].

The phenomenon of lossy-mode-resonance (LMR), being the physical basis of operation of a specific class of sensors, results from the fact that for certain thickness and optical properties of the dielectric overlay the light in the thin-film-coated fiber core experiences wavelength-dependent attenuation, i.e., in the optical spectrum characteristic transmission minima appear [43]. The effect is a consequence of coupling between core modes and specific lossy modes of the overlay. The condition of appearance of LMR is that the real part of the overlay permittivity is positive and larger in magnitude than both its own imaginary part and the real part of permittivity of the materials surrounding the overlay, i.e., of the fiber core and an external medium. The crucial issue for sensing applications is that spectral positions of the minima depend also on refractive index of the surrounding medium (external RI). This fact enables the use of LMR structures as refractometers. On top of it, the sensor surface functionalization allows also for biosensing applications of these sensors [44]. It must be noted that the spectral position of the resonant wavelengths and the device sensitivity can be fine-tuned just by adjustment of the thickness and optical properties of the overlay.

The most often used for the purpose of overlay for LMR-based fiber sensors are oxides of aluminium (Al_2O_3) [45], titanium (TiO_2) [46,47], tin (SnO_2) [48], indium (In_2O_3) [49], or indium doped tin oxide (ITO) [50–55]. Also silicon nitride (Si_xN_y) [56], diamond-like carbon [57] and polymeric overlays, like polyallylamine hydrochloride/polyacrylic acid (PAH/PAA) [58] have already been used. However, the overlays on the fiber sensors have been deposited in the form of single layers only, i.e., just one material at a time has been used for formation of an individual overlay. The possible reason for this can be a complicated numerical modelling of such a sensor, but also a number of technological difficulties. According to our best knowledge only once multilayer LMR-based structure was discussed theoretically, but the concept has never been practically implemented [59].

In this paper we present capability for tuning the properties of optical fiber LMR sensors by using the ALD-based dielectric overlays. We show the possibility of tuning its sensing properties by ALD-deposited single-layer overlays, and also we put into practice the concept of double-layer dielectric overlays for the first time. Such overlays comprise two layers, each deposited by using different technique, namely ALD and plasma-enhanced CVD (PECVD). The presented solution offers to the field of thin film optical fiber sensors technological flexibility available thanks to application of the ALD technique.

2. Experimental details

2.1. Numerical modelling

The numerical model used for the simulations of the LMR phenomenon has already been described elsewhere [57]. The aim of the performed simulations is to show the effect of variation in the LMR response when properties of the overlays change and to determine the range of the properties where the effect can be seen. Wavelength-dependent response of the structures to variations in

external RI was normalized using spectrum obtained for a bare sample surrounded by air.

2.2. The fibers and their preparation before deposition of the overlays

For preparation of the sensors polymer-clad silica (PCS) multi-mode optical fibers with 400 μm core diameter were used. While total length of the fiber samples was up to 150 mm the only 25 mm-long central section of each fiber was stripped mechanically and chemically from outer polymer coating and cladding [57]. Then the fiber samples were extensively cleaned with isopropanol.

2.3. Deposition of the overlays by ALD

The metal oxides were deposited by ALD using thermal mode at the thermocouple-controlled reactor temperature of 100 ± 0.1 °C, in Beneq TFS-200–190 system. Such low temperature of the process was unavoidable because of the thermal vulnerability of a polymer coating that majority of optical fiber length is originally covered by. Additionally, low temperature enables deposition of amorphous films and hence smoother surfaces than for also available for ALD polycrystalline forms. Amorphous films are preferred due to more uniform film thickness distribution as well as for suppression of light scattering. The carrier/purging gas was 6N-purity argon (Ar) flowing through the reactor with constant rate of 1000 sccm that generated the pressure inside the reactor of approx. 2 mbar. As the oxygen chemical precursor was used deionized water ($\text{DI H}_2\text{O}$) kept at 19 ± 0.1 °C in Beneq's standard LS container. Tantalum pentachloride (TaCl_5 , kept at 85 ± 0.1 °C), tetrakis(ethylmethylamino)zirconium (TEMAZ, kept at 75 ± 0.1 °C), and tetrakis(ethylmethylamino)hafnium (TEMAH, kept at 78 ± 0.1 °C), all delivered by Volatec Oy and kept in Beneq's standard HS 300 containers, were used as chemical precursors for tantalum, zirconium and hafnium, respectively.

The precursor exposure times, that are responsible for substrate-surface-saturation conditions for all complementary half reactions of chemisorption involved in the employed ALD processes, were experimentally determined before the experiments described in this paper; the crucial role of exposure of complementary precursors and surface saturation in ALD reactions has already been explained, e.g., in Ref. [60,61]. They were studied using Si test substrates, and they are not described here. On the basis of these experiments the appropriate precursor/purge-cycling schemes were determined. For the optimized cycling conditions used in this work the growth per cycle (GPC) measured for Si-based samples was approx. 0.133, 0.150 and 0.145 nm/cycle for the Ta_xO_y , ZrO_2 and HfO_2 films, respectively. The film growth rate was approx. 0.019, 0.022, and 0.021 nm/s for the Ta_xO_y , ZrO_2 and HfO_2 , respectively. It should be noticed here, that generally the composition of as-deposited Ta_xO_y may vary and contain compounds of tantalum with various oxidation states between +2 and +5, i.e., TaO , Ta_2O_3 , TaO_2 and Ta_2O_5 . The thickness range of the ALD-based layers investigated in this paper was 201–211 nm for the HfO_2 , 216–244 nm for ZrO_2 , and 50 nm for the Ta_xO_y .

Before entering the ALD reactor the fibers were rinsed extensively with acetone and isopropanol at the ambient temperature (approx. 20 °C). For each ALD process three fibers were introduced into the reactor and they were positioned in a run-to-run-repeatable arrangement.

2.4. Deposition of the overlays by PECVD

The Si_xN_y thin films were deposited on the fiber samples using Oxford Plasmalab 80 Plus system. The fiber samples were placed in the plasma reactor on U-type holder on height of approx. 5 mm over the electrode. The reference Si wafers were placed next to

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