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Regular Articles Toluene optical fibre sensor based on air microcavity in PDMS

Daniel Kacik*, Ivan Martincek

Department of Physics, University of Zilina, Univerzitna 1, 01026 Zilina, Slovakia

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1. Introduction

Optical fibre sensors have unique properties such as small size, light weight, high sensitivity, biocompatibility, corrosion resistance, immunity to electromagnetic interference, continuous measurements and in-situ monitoring of chemical parameters in industrial processes [1]. The sensors can work in different ways. One of the most sensitive ways is sensing measurands by the interference of light. Configuration of the interferometer depends on different parameters. Usually the interferometer consists of a set of mirrors dividing the light into arms, and then decoupled back to compare the phase of travelled light in particular arms. One interesting aspect of mirror creation is offered by a Fabry-Perot interferometer (FPI). The mirrors that form the Fabry-Perot cavity in fibre optics can be reflective splices between two identical optical fibres [2], the section of photonic crystal fibre between singlemode fibres [3] or etched polymer fibre with Bragg grating [4]. FPIs can be built not only as all-fibre (intrinsic), but also as extrinsic sensors. In the case of extrinsic sensors the typical FPI sensor is built between a cleaved optical fibre end and an elastic diaphragm, such as a polymer diaphragm [5], silica diaphragm [6] or metal diaphragm [7].

Sensors based on FPI have a wide range of applications similar to other optical-fibre sensors based on interference, such as temperature measurement, refractive index, pressure, transverse load, etc. [8].

ABSTRACT

We prepared and demonstrated a compact, simple-to-fabricate, air microcavity in polydimethylsiloxane (PDMS) placed at the end of a single-mode optical fibre. This microcavity creates a Fabry-Perot interferometer sensor able to measure concentrations of toluene vapour in air. Operation of the sensor is provided by diffusion of the toluene vapour to the PDMS, and the consequent extension of length *d* of the air microcavity in PDMS. The sensor response for the presence of vapours is fast and occurs within a few seconds. By using the prepared sensor toluene vapour concentration in air can be measured in the range from about 0.833 g.m⁻³ to saturation, with better sensitivity than 0.15 nm/g.m⁻³ up to maximal sensitivity 1.4 nm/g.m⁻³ at around concentration 100 g.m⁻³ in time 5 s.

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Recently, fibre-tip sensors with microcavities have been proposed. For example, microcavities fabricated by using a fusion splicer and pressurizing gas chamber [9], or applying polymer at the end of a small segment of photonic bandgap fibre [10]. The sensors were proposed in order to detect pressure, but when a polymer with favourable physicochemical properties is used, a similar configuration can also be used for the detection of organic volatile compounds (for example, toluene).

The sensing of volatile organic compounds is of importance in a range of applications, for example, monitoring air quality in both indoor and outdoor environments. Volatile organic compounds can originate from fuel and petroleum products, from paints and by combustion processes, natural sources and farming. Human exposure to these chemicals, even at low concentration, can be hazardous due to producing short- and long-term adverse health effects.

Sensitivities of particular configurations of sensors for different organic chemicals are different. A sensor based on a surface relief D-shaped fibre Bragg grating had sensitivity to acetone: $-1.65 \cdot 10^{-4}$ pm/ppm from concentration app. 6000 ppm [11]. For zeolite, a thin film-coated fibre sensor based on FPI, the sensitivity was $4.28 \cdot 10^{-3}$ nm/ppm when the concentration of isopropanol ranged from 350 ppm to 2450 ppm [12]. In Ref. [15] the proposed sensor is based on long period grating coated with calixarene to form the nanostructured coating which achieved sensitivity to toluene 231 ppmv (ppm in volume) with spectrometer resolution of 0.3 nm and recovery time of order of 15 s.

In this article, we report a preparation of polydimethylsiloxane (PDMS) FPI located at the end of a single-mode optical fibre (SMF). A fibre tip is treated by applying thin paraffin wax film to the fibre







 ^{*} Corresponding author.
E-mail addresses: daniel.kacik@fel.uniza.sk (D. Kacik), ivan.martincek@fel.uniza.
sk (I. Martincek).

end. Then the fibre end is packed with PDMS. After curing, the fibre end is heated to diffuse wax to the PDMS. This reduces the adhesion and allows the creation of the microcavity between the fibre end face and the PDMS layer. Then the length of microcavity is fixed by covering it with a second layer of PDMS.

Based on toluene influence we investigated the function of the FPI fibre. When toluene is applied to such a FPI, the length of the microcavity will change. So we determine the sensor response on toluene concentrations in air. The main advantages of the developed sensor are its compactness and its very short analytical time (response and desorption time), which was found to be 5 s.

2. Operational principle

The PDMS microcavity placed at the end of the SMF forms an extrinsic optical fibre FPI. The interferometer consists of two mirrors with reflectivity R_1 and R_2 , respectively. The mirrors are separated by distance *d*. In our case, the first mirror of the interferometer is formed by the surface S_1 with reflectivity R_1 at the interface SMF/air, and the second mirror is formed by the surface S_2 with reflectivity R_2 at the interface air/PDMS at the end of the microcavity. SMF is used as input/output fibre of the interferometer. It is shown schematically in Fig. 1.

If the light with intensity I_0 is launched to the SMF, then the surface S_1 will reflect part I_1 of the light, and the rest will be transmitted. A similar effect will take place over an area S_2 that reflects the intensity I_2 . Light reflected from both interfaces will be coupled back into the SMF core and interfere with each other. In our case, as the reflectance of the surfaces S_1 and S_2 is low, multiple reflections can be neglected and the intensities I_1 and I_2 can be written as $I_1 = R_1I_0$ and $I_2 = (1 - R_1)R_2I_0$, respectively. So the intensity I_R of the light reflected from the air microcavity can be described as

$$I_R = I_1 + I_2 + 2\sqrt{I_1 I_2} \cos\left(\frac{4\pi d}{\lambda}\right),\tag{1}$$

where *d* is the length of air microcavity in PDMS and λ is wavelength of used light in a vacuum.

It is known that liquid and gaseous toluene is well infiltrated into the PDMS, causing increases in volume [13] also like other organic volatile compounds. In our case, diffusion of toluene vapour to the PDMS caused increases of the air microcavity length. Thus, it results in an optical phase shift of the reflected intensity I_2 . The optical phase shift in the PDMS Fabry-Perot interferometer at constant temperature and constant pressure depends on the time of influence, and on the toluene vapour concentration in the PDMS.



Fig. 1. Schematic of the extrinsic optical fibre Fabry-Perot interferometer consisting of single-mode optical fibre (SMF) as input/output fibre and two mirrors with surfaces S_1 and S_2 separated by distance *d*.

3. Fabrication of air optical microcavity in PDMS

For the fabrication of the air microcavity in the PDMS placed at the end of the optical fibre PDMS Sylgard 184 (Dow Corning) was used, which was supplied as two-part liquid component kits. PDMS was prepared by mixing prepolymer (part A) and curing agent (part B) in the ratio 10:1. PDMS possess properties such a hydrophobility, hydrolytic stability, non-flammable, high chemical stability, optically clear and its refractive index is close to that of glass [14]. The fabrication technological process of the air microcavity can be described as follows: after removing the primary fibre coating on the cladding at the end of the single-mode optical fibre a thin paraffin wax film was applied. However, it wasn't applied to the cleaved face of the SMF. The paraffin wax film had a thickness of approximately 20 µm, and covered the fibre for a length of about 370 µm. Then the first layer of PDMS in a teardrop shape was applied to the end of the fibre up to the distance where the wax was. Also, the PDMS was applied to the face end of the SMF (Fig. 2A). After curing the fibre end with PDMS, the coating was heated to 70 °C for 60 min. During heating the paraffin wax changed to a liquid state. During this time, liquid paraffin wax diffused into the PDMS. As a result, the adhesion between the PDMS and the cladding of SMF was reduced. Then, at room temperature the PDMS laver was moved and between the fibre end and PDMS the air optical microcavity was created (Fig. 2B). The length of microcavity *d* was controlled by measurement of interference pattern and set to 70 µm. In order to fix the length of the optical microcavity at the end of the fibre, the first PDMS layer was encapsulated by a second layer of PDMS (Fig. 2C). The second layer of PDMS covered



Fig. 2. Technological process of preparation of air optical microcavity in polydimethylsiloxane (PDMS) located at end of single-mode fibre (SMF).

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