

# Concentration sensor based on a tilted fiber Bragg grating for anions monitoring



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## ABSTRACT

The ubiquity and importance of anions in many crucial roles accounts for the current high interest in the design and preparation of effective sensors for these species. Therefore, a tilted fiber Bragg grating sensor was fabricated to investigate individual detection of different anion concentrations in ethyl acetate, namely acetate, fluoride and chloride. The influence of the refractive index on the transmission spectrum of a tilted fiber Bragg grating was determined by developing a new demodulation method. This is based on the calculation of the standard deviation between the cladding modes of the transmission spectrum and its smoothing function. The standard deviation method was used to monitor concentrations of different anions. The sensor resolution obtained for the anion acetate, fluoride and chloride is  $79 \times 10^{-5}$  mol/dm<sup>3</sup>,  $119 \times 10^{-5}$  mol/dm<sup>3</sup> and  $78 \times 10^{-5}$  mol/dm<sup>3</sup>, respectively, within the concentration range of  $(39\text{--}396) \times 10^{-5}$  mol/dm<sup>3</sup>.

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## 1. Introduction

Considering that anions play an important role in biological and environmental applications, the development of sensors for anion monitoring is of a great interest by the scientific community. Among different anions, acetate (CH<sub>3</sub>CO<sub>2</sub><sup>−</sup>), fluoride (F<sup>−</sup>) and chloride (Cl<sup>−</sup>) are of extreme importance in various situations. Acetate is present in numerous metabolic processes [1] as well as can be an indicator of organic decomposition in marine sediments [2]. The monitoring of fluoride in water is important to prevent dental caries [3] and its detection can help in clinical treatments such as osteoporosis [4]. With regard to chloride, a small change of the anion flow across the cell membranes can be an indicator of a disease named cystic fibrosis [5]. From this short list, it is notorious that the monitoring of anions is highly desirable.

Anions sensing is traditionally performed by host molecules that are designed to recognize a target anion within a given concentration range [6,7]. When the binding between the host and the anion occurs, the host molecule emits a signal which can be

either colorimetric [8], fluorescent [9] and/or an electrochemical response [10].

In the last decade, the development of optical fiber sensors for the detection of chemical compounds has become an interesting topic of investigation due to the possibility of producing small sized sensors that are able to remotely and continuously operate in contact with the sample.

A number of sensor configurations have been proposed for detection of chemical compounds by monitoring the change of the surrounding refractive index (SRI). These include tapers [11], fiber Bragg gratings (FBGs) [12,13], tilted fiber Bragg gratings (TFBGs) [14], long period gratings (LPGs) [15,16] and surface plasmon resonance (SPR) sensors [17]. Fiber tapering has demonstrated high sensitivity to the SRI; however, these sensors are significantly weaker than unmodified fiber due to the reduction of the fiber diameter [11]. FBGs can also be used to measure SRI by etching the cladding, which leads to a decrease of the fiber mechanical strength [12,13]. LPGs are sensitive to the SRI due to the coupling of radiation from the propagating core mode to the forward-propagating cladding modes [18]. The most prominent drawback of LPGs is the high sensitivity to other parameters such as temperature and strain, which can cause undesirable effects as an SRI sensor. SPR sensors exhibit very high sensitivity to the SRI; however, these sensors normally operate in the visible region which presents higher optical losses compared to the infrared region [17].

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A TFBG consist of a periodic modulation tilted with respect to the longitudinal axis of the fiber core which enables the coupling between the forward-propagating core mode and the forward/backward-propagating cladding modes [19]. The core and cladding modes appear as a series of multiple dips in the transmission spectrum. The resonance wavelength and the intensity of the dips change as a function of the SRI. Different demodulation methods can be employed to determine the changes of the SRI in a TFBG [20]. Initially, Laffont and Ferdinand reported a technique based on the envelope area of the transmission spectrum [21]. Later, Caucheteur and Mégret improved this method through the correlation between the transmission spectrum and the calculation of two statistical parameters, namely Skewness and Kurtosis [22]. Another widely used method is based on the calculation of the transmission spectrum area [23,24]. In 2007, Chan et al. reported a method in which the wavelength separation between selected cladding modes was calculated [25]. More recently, an alternative demodulation method based on the measurement of the single wavelength time delay has been exploited [26].

In this paper, the use of a TFBG as a concentration sensor for three different anions species namely  $\text{CH}_3\text{CO}_2^-$ ,  $\text{F}^-$  and  $\text{Cl}^-$  is reported. The detection of anions is performed by measuring the changes of the SRI caused by different anion concentrations. For this purpose a new demodulation method based on the calculation of the standard deviation between the transmission spectrum of a TFBG and its smoothing function was developed.

## 2. Theory of TFBG

TFBGs are classified as short period gratings whose refractive index modulation is tilted with respect to the longitudinal axis of the fiber. TFBGs written with a small tilt angle promote the coupling of the forward-propagating core mode to the backward-propagating cladding modes and decrease the coupling to the backward-propagating core mode [19]. As a consequence, a number of resonances of the cladding modes as well as the resonance of the core mode can be observed in the transmission spectrum (Fig. 1).

The resonances can be explained by the phase match condition through the following equations:

$$\lambda_B = \frac{2n_{\text{eff,core}}\Lambda_T}{\cos(\theta)} \quad (1)$$

$$\lambda_{\text{clad}}^i = \frac{(n_{\text{eff,core}} + n_{\text{eff,clad}}^i)\Lambda_T}{\cos(\theta)} \quad (2)$$

where  $n_{\text{eff,core}}$  and  $n_{\text{eff,clad}}^i$  are the effective refractive indices of the core mode and the  $i$ th cladding mode, respectively. The nominal grating period is represented by  $\Lambda_T$  and can be expressed as  $\Lambda \cos(\theta)$ , where  $\Lambda$  is the pitch along the fiber axis and  $\theta$  is the tilt angle.

The effective refractive index of the cladding modes depends on the SRI. Therefore, a change of the SRI affects the transmission spectrum of a TFBG. When the grating is immersed in a solution with a small SRI, numerous resonances are visible as a result of the coupling to the backward-propagating cladding modes. As the SRI increases, the cladding modes are gradually cutoff from lower to higher wavelengths and the transmission spectrum gradually approaches its smoothing function. When SRI equals  $n_{\text{eff,clad}}^i$ , the cladding modes are no further guided and a smoothed spectrum is observed.

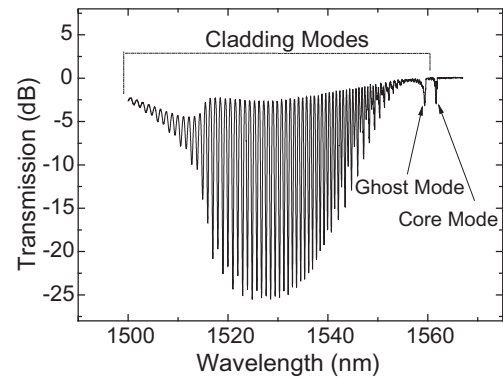


Fig. 1. Transmission spectrum of a tilted fiber Bragg grating.

## 3. Experimental methods

A TFBG was fabricated with a 248 nm KrF excimer laser in a standard single mode photosensitive fiber (PS1250/1500, Fibercore), using the phase mask technique. The fiber was previously hydrogen-loaded at 150 bars during two weeks to further enhance its photosensitivity. The phase mask, with a period of 1073.11 nm, was tilted in the perpendicular plane to the laser beam. The grating was written with a total length of 15 mm and an internal tilted angle of 8°. After the inscription, the grating was placed in a heat chamber at 80 °C for 24 h to lose the excess of hydrogen.

The experimental setup used for anions monitoring is presented in Fig. 2.

The grating was placed in a cylindrical glass container (reactor) and both sides of the fiber were fixed, one at a holder and the other at a manual linear stage. All experiments were performed under a temperature of 23.0 °C controlled by a climatic chamber (Challenge 340, Angelantoni Industrie) with a resolution of 0.1 °C. The manual linear stage was used to keep the fiber taut and the magnet was introduced in the reactor to ensure the homogeneity of the solutions. The transmission spectrum of the TFBG sensor was acquired by an Optical Network Analyzer (ONA) with a wavelength resolution of  $4.2 \times 10^{-3}$  nm.

The TFBG was characterized to the SRI by using different concentrations of glycerin solutions, providing a refractive index range from 1.341 to 1.437 RIU (refractive index unit). The refractive index values were measured at ambient temperature by a commercial Abbe refractometer (AR4D, Krüss Optronic), at the wavelength of 589 nm and with a resolution of  $1 \times 10^{-3}$ . The tests were performed by acquiring the transmission spectrum of the TFBG when immersed in each glycerin solutions, starting from the lowest to highest refractive index. After the first set of glycerin

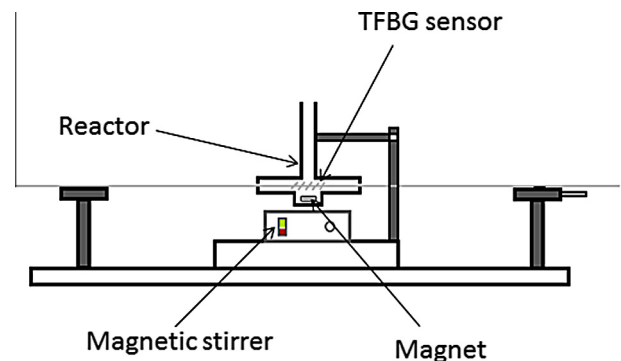


Fig. 2. Experimental setup for anions monitoring.

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