

# Research Article High Sensitivity Refractive Index Sensor by D-Shaped Fibers and Titanium Dioxide Nanofilm

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This paper presents a high sensitivity liquid refractive index (RI) sensor based on lossy mode resonance (LMR) effect. The D-shaped fibers coated with nanosized titanium dioxide ( $TiO_2$ ) thin film as a sensing head were submerged into different refractive index solutions. The variations in the optical spectrum of the proposed RI sensor with different refractive index solutions. The results show that the optical spectrum peaks shifted towards the longer wavelength side with increasing the refractive index. For the proposed fiber sensing head with a polishing residual thickness of 72  $\mu$ m, the maximum shift of the absorption peak was 264 nm. The sensitivity of the proposed RI sensor was 4122 nm/RIU for the refractive index range from 1.333 to 1.398.

# 1. Introduction

Many fiber-optic sensors for refractive index (RI) sensing have been developed due to some advantages such as small size, high sensitivity, light weight, and immunity to external electromagnetic interference. Various fabrication methods for fiber-based RI sensors have been reported in the literature [1], including surface plasmon resonance (SPR) [2, 3], evanescent field [4], fiber gratings [5, 6], and optical fiber interferometry [7]. However, the sensitivity of the traditional sensing techniques needs to be improved in practical applications. Iadicicco et al. [5] reported an etched fiber Bragg grating RI measurement, while etching can also be applied to a long period of grating to increase sensitivity to external refractive indices [6]. Optical fiber sensors based on evanescent wave generate a strong interaction between the guided wave and the surrounding materials. However, most of the reported sensors showed a low-sensitivity or low-detection range. To overcome this problem, we proposed a D-shaped optical fiber coated with a high refractive index titanium dioxide  $(TiO_2)$  nanofilm to enhance the sensitivity and to extend the detection range of the sensor.

In the last decades, thin film coated onto a fiber-optic has become a widely explored technique in the field of sensors. The effects of depositing a thin, highly refractive index (RI) layer onto the cladding over the grating region have been reported [8, 9]. Since titanium dioxide  $(TiO_2)$  is one of the highly refractive index materials, TiO<sub>2</sub> is known to be a good photocatalytic material under UV radiation. In this study, we present the fabrication of high sensitivity refractive index fiber-optic sensors based on D-shaped fibers and nanosized  $TiO_2$  coatings. From the optical point of view,  $TiO_2$  nanofilm deposited on D-shaped fibers can generate lossy mode resonance (LMR) effect under particular conditions [10]. In this work, we fabricate a new liquid refractive index fiber-optic sensor called a D-LMR (D-shaped fiber with lossy mode resonance) sensor that combines with the D-shaped fibers and nanosized TiO<sub>2</sub> coatings to achieve a high sensitivity sensor with a wide range of liquid refractive indices. We also utilized a DC magnetron sputtering technique to deposit TiO<sub>2</sub> nanofilms on the polished surface of D-shaped fibers for the fabrication of liquid refractive index fiber sensors. The proposed D-shaped fiber sensor structure includes a low index upper cladding (about  $3-5 \mu m$  thick) between the fiber core and the TiO<sub>2</sub> layer that is different from an uncladded fiber structure in the literature [10-12]. Our fiber structure design will help in giving the D-shaped fiber good mechanical support after side-polishing. This paper demonstrates the D-LMR type RI sensors with a high sensitivity for liquid refractive index measurements. The characteristics of the resonance-based fiber-optic sensors could be extensively used as refractometers to detect the refractive index changes in liquids.

## 2. Method

If a side-polished optical fiber is coated with a thin film, the propagation of the guided wave is affected. The thin film coatings combined with side-polished optical fibers can produce two different resonance types: one is surface plasmon resonances (SPRs) [8] and the other is lossy mode resonances (LMRs) [9-11]. The surface plasmon resonance (SPR) 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 films. The LMR effect can be generated by the metal oxide films whose real part of the permittivity is positive and higher in magnitude than both the imaginary part of the permittivity and the permittivity of the surrounding material. The LMR effect is achieved for metal oxide films with low imaginary part of the complex refractive index. In general, a specified thin film coated onto a D-shaped fiber can generate LMR effect based on a modified Kretschmann configuration [10]. LMR phenomenon has been observed with metal oxides, such as indium tin oxide (ITO) [12], titanium oxide [13], and indium oxide [14]. Apart from this, LMR effect appears for both TM and TE polarized light, and the generation of multiple resonances without modifying the optical fiber geometry is also possible [15]. According to the mode coupling theory, an LMR is the result of light coupling between evanescent waves and lossy modes guided in the absorbing thin films. Thus, the analysis of the evolution of the modal effective refractive index is helpful in understanding the generation of the resonance.

Since all of the materials are characterized by their relative permittivity, titanium dioxide thin films can be characterized by a complex form of relative permittivity. The oscillatory model represents the dielectric constant of  $TiO_2$  thin films and it can be expressed as follows [16]:

$$\varepsilon(\omega) = \varepsilon_{\infty} - \frac{\omega_p^2}{\omega^2 + i(\omega/\tau)},\tag{1}$$

where  $\varepsilon_{\infty}$  is the dielectric constant of the high electron density region in the film,  $\tau$  is the Drude damping time for free electrons,  $\omega$  is the incident light frequency, and  $\omega_p$  is the plasma frequency. It should be noted that the dielectric constant was predicted by the Drude model [17]. The plasma frequency is determined by the following formula:

$$\omega_p^2 = \frac{Ne^2}{\varepsilon_0 m^* m},\tag{2}$$

where *N* is the electron density, *e* is the electron charge,  $\varepsilon_0$  is the dielectric constant, *m* is the electron mass, and  $m^*$  is the optical effective mass for carriers [18].

As mentioned above, the generation of LMR is based on D-shaped fibers coated with absorbing thin films which modified the well-known Kretschmann configuration. If TiO<sub>2</sub> films are deposited on the polishing surface of D-shaped fibers, then the real part of the refractive index of TiO<sub>2</sub> films will always be higher than that of the D-shaped fiber in the wavelength region of our study. Besides, when the index of the surrounding medium above the TiO<sub>2</sub> nanolayer is 1.333, the structure will support guided modes. These modes will be lossy because of the imaginary part of the index of TiO<sub>2</sub> films. Thus, the dispersion curves for the proposed sensor structure (D-shaped fiber/TiO<sub>2</sub> layer/surrounding medium) will be similar to the dielectric waveguides [19]. The number of modes supported by this waveguide increases with increasing the film thickness. Batchman and McWright [20] studied the light propagation through semiconductor cladded waveguides. The attenuation maxima of the light propagating through the optical waveguide can be obtained for specific thicknesses and at certain wavelengths. This effect can be explained as a periodic coupling between the guided modes of the lossless structure and the lossy modes supported by the high refractive index material. Therefore, the mode loss is maximal if the thickness of the coating layer corresponds to the cut-off thickness for that particular mode. Carson and Batchman [21] reported that a thin film is deposited on the single mode waveguide; the mode losses for either TE or TM become maximal at its particular thickness. This happens due to the lossy nature of the thin film and the phase matching between the guided mode supported by a lossless dielectric waveguide and the lossy mode supported by the thin film.

In this work, the proposed D-LMR fiber sensor can be fabricated by using the D-shaped fiber and thin film coating technique. It is a challenging work to keep a uniform thickness of the TiO<sub>2</sub> nanolayer coated on a side-polished surface of the single mode fibers. A DC magnetron sputtering technique is an efficient approach to obtain a uniform nanolayer coating of TiO<sub>2</sub> films with a low surface roughness. The performance of the proposed RI sensor is evaluated in terms of wavelength sensitivity and linearity.

#### 3. Experimental

Using a side-polishing technique to make D-shaped fiber, we modified the motor-driven polishing method [22] to suspend the optical fibers and to control the polishing surface depth. To ensure correct polishing depth, a PC-based measuring system with a photometer was used to monitor and control the polishing process in real time. A large increase of power loss during polishing indicates that the cladding is thin enough for the light to radiate from the fiber core and that the polishing should be stopped.

A high sensitivity fiber-optic sensor is reached by thinning the optical fiber enough to obtain an evanescent wave interaction of the guided light with the surrounding mediums. For D-shaped optical fibers, the core is very close to the flat surface of the structure. As a direct consequence, an optical fiber thinning is enough to allow evanescent wave interaction of the guided light with the surrounding mediums. The D-shaped fiber section is sensitive to the surrounding medium refractive index allowing the measurement of different refractive indices in liquids.



FIGURE 1: Schematic diagram of optical fiber polishing arrangement.

A single mode fiber (SMF-130V) was used to fabricate the sensing devices. Before the thin film deposition, the cladding was partially removed, and the resultant portion was ultrasonically cleaned. After this cleaning process, the fabrication of the sensors involved two different steps. First, we used the single mode fiber, ground/polished by using a home-made side-polishing machine. During the process of polishing the fiber, we need to monitor the polishing depth by checking the transmission light power levels, as shown in Figure 1. This measurement can confirm when the polished surface reaches the near fiber core region. The polished length was altered by changing the contact angle of the optical fiber on the wheel or by using a polishing wheel of different diameter. Under the optical fiber polishing conditions, where the wheel rotated at 15 rpm, the tension force on the optical fiber was approximately 0.2-0.3 N and the wheel was lubricated with liquid paraffin; the radial force on the wheel from the optical fiber was essentially uniform over the contact length and the optical fiber was polished uniformly over that length. The polished fiber diameter was also further measured by an optical microscope to precisely obtain the polished depth. The thickness and polishing length of the side-polished fibers were also measured by using an optical microscope. Figure 2 shows the microscopic image of the D-shaped fiber. The residual thickness of the side-polished fiber was about 72  $\mu$ m (including a 5  $\mu$ m-thick cladding layer), and the polishing length of the D-shaped fiber was 30 mm. Then, TiO<sub>2</sub> nanofilm was deposited on the D-shaped fiber surface by using a DC magnetron sputtering system. TiO<sub>2</sub> has the advantages of nontoxicity, environmental compatibility, high refractive index, and low price; therefore, we choose TiO<sub>2</sub> as the coating material.

Prior to the deposition, the coating substrates (including silicon wafers and the D-shaped fibers) had been ultrasonically cleaned in acetone and ethanol and then had been dried by a dryer. In a high-vacuum sputtering chamber, D-shaped fibers and silicon wafer were mounted onto a substrate holder 200 mm in diameter that rotated at a speed of 30 rpm. The flat side of D-shaped optical fibers offers a unique substrate on which thin films can be deposited. The D-shaped optical fibers were embedded in V-shaped



FIGURE 2: Microscopic image of the D-shaped fiber.

groove of a special fiber holder and the flat side of D-shaped fibers was parallel mounted to the surface of the substrate holder to keep the coating thickness uniform. For the DC sputtering depositions, titanium targets with purity of 99.99% and diameter of 76.2 mm were used. The target to substrate distance was 120 mm, the total pressure being set at  $1.5 \times 10^{-3}$  Torr. The reactive gas partial pressure was kept constant at  $4.5 \times 10^{-4}$  Torr during the deposition. For the preparation of the nanosized TiO<sub>2</sub> films, a mixture of argon and oxygen gases was used. The DC power and heating temperature were controlled to be 100 W and 80°C, respectively. Thin film was simultaneously deposited on the silicon wafers and the side-polished surfaces of the single mode fibers (SMF). The TiO<sub>2</sub> film's thickness of 84 nm was measured by using an ellipsometer.

To characterize the optical response of the D-LMR sensing device, a typical transmission setup was used. A portion of the coated optical fiber was cleaved at both ends and spliced to optical fiber patch cords. The spectral response of the sensor was obtained by using a typical optical transmission setup. Figure 3 shows that this setup consisted of a halogen white light source (Ocean Optics, HL-2000) connected at the input of the optical fiber, and the other end was attached to a nearinfrared spectrometer (Ocean Optics, NIR 512). Thus, the light coupled to the side-polished fiber passed through the cladding removed sensitive region, which is located in the optical transmission pathway.

## 4. Results and Discussion

The absorption peaks generated by the LMR phenomenon can be observed in optical transmission spectrum. The wavelength interrogation method along with D-LMR type optical fiber sensors was considered in our analysis. The LMR wavelengths were determined from the normalized transmittance versus wavelength plot for different sensing liquid refractive indices. The data analysis for the proposed sensor was done by using MATLAB® software. The different sets of sensing medium (NaCl solutions) refractive indices



FIGURE 3: Schematic of the proposed RI sensor and experimental setup.

0.30



FIGURE 4: LMR spectra of the D-shaped fibers coated with  $TiO_2$  nanofilm for different liquid refractive indices.

0.25 [ransmittance (%) 0.20 0.15 0.10 0.05 0.00 1000 1100 1200 1300 1400 1500 1600 1700 900 Wavelength (nm) 1.333 1.366 - 1.338 1.382 - 1.356 1.398

FIGURE 5: The transmittance spectra of different refractive index solutions by D-LMR optical fiber sensor.

used for the analysis were 1.333, 1.338, 1.356, 1.366, 1.382, and 1.398. Figure 4 shows the LMR transmission spectra of D-shaped fibers coated with nanosized  $TiO_2$  film for different liquid refractive indices. The sensing sensitivity is defined as the shift of resonance wavelength versus the change in surrounding refractive index. If the refractive index value is increased by higher concentration of NaCl solutions, the LMR peak shifts to higher wavelengths. From the transmittance spectra, when the refractive index of the external medium is 1.333, the absorption peak is located at 1307 nm. When the refractive index of the external medium is 1.338, the absorption peak is located at 1335 nm. When the RI is 1.382, the peak is at 1520 nm, and when its value is 1.398 the peak reaches 1571 nm. Figure 5 illustrates the normalized transmittance of D-LMR optical fiber sensor for different

refractive index solutions. Here the transmittance is defined as the ratio of the transmitted light intensity to the incident light intensity. It is important to note that the wavelength's shift of the resonance peaks is a function of the refractive index of NaCl solutions, as shown in Figure 6. The sensing sensitivity of the D-LMR sensor was 4122 nm/RIU; RIU is refractive index units, for the fiber residual thickness of 72  $\mu$ m and the polishing length of 30 mm. Figure 6 also shows the linear fitting of the resonant wavelength as a function of the liquid refractive index. The linear fitting curve demonstrates  $R^2$  value of 0.980. In our previous publication [23], a liquid refractive index sensor using double-sided polishing long period fiber grating (DSP-LPFG) was reported. A sensing sensitivity of 143.4 nm/RIU for the DSP-LPFG sensor was obtained from the RI range of 1.333–1.375. In this work, the



FIGURE 6: LMR wavelength as a function of the refractive index for different NaCl solutions.

experimental results show that the sensing sensitivity of the D-LMR sensor is 28 times higher than that of the DSP-LPFG sensor. These results also show that the sensing sensitivity of the D-LMR sensor is about 12 times higher than that of a tilted-LPFG sensor for liquid refractive index measurements [6]. The detection range of the proposed RI sensor is also extended.

#### 5. Conclusion

We have proposed and demonstrated a novel D-LMR fiber sensor by coating  $TiO_2$  nanofilms on D-shaped fibers. The D-LMR sensor response to the liquid refractive index changes has been carried out by measuring the resonance wavelength's shift. The results show that LMR peaks shift to higher wavelengths with increasing the liquid refractive index. The greatest sensitivity of the RI sensor that has been experimentally demonstrated is 4122 nm/RIU for the RI range from 1.333 to 1.398. Compared with other sensors, we propose that a simple structure based on a D-shaped fiber and nanosized  $TiO_2$  coating layer will be more sensitive for the liquid refractive index measurements with the advantages of being more compact, of low cost, and easily portable. The general idea of proposed D-LMR sensor can also be applied to other sensing applications.

## **Conflicts of Interest**

The authors declare that they have no conflicts of interest.

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