Spatial frequency spectrum of SPR-TFBG: A simple spectral analysis for in-situ refractometry

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ARTICLE INFO

Keywords:
Surface plasmon resonance (SPR)
TFBG sensor
Refractometry
Spatial frequency spectrum

ABSTRACT

Surface plasmon resonance-tilted fiber Bragg gratings (SPR-TFBG) are known for their ultra-high sensitivity to various liquid measurands. However, the deposition angle of the gold film on TFBG being one of the critical fabrication parameters remains ill-explored. This fabrication parameter has a strong influence on the some of the optical spectral properties such as the cut-off wavelength, surface plasmon resonance (SPR) wavelength and attenuation. It affects the choice of selection of the detection parameters and subsequently the performance of detection. In this study, we perform an intensive study on the spatial frequency spectrum, generated from the optical spectrum based on Fast Fourier Transform (FFT). It is found that some of the features in the optical spectrum such as the SPR dip and cut-off properties are inherited in the frequency spectrum. The influence of deposition angles can be observed from the frequency spectra. The greatest advantages of the frequency spectrum are the simple spectral curve that enables easy identification and extraction of the frequency parameters fC and fSPR that have a linear relationship with the surrounding refractive index (SRI). Lastly, we demonstrated the use of fSPR for in-situ refractometric measurement of a saline solution with a varying concentration. This successful demonstration has proven the feasibility of this novel interrogation technology for real-world applications.

1. Introduction

Rapid human modernization and exponential growth in population result in an urgent need in the development of SPR-TFBG sensors for detection, diagnosis, and determination in the fields of food and water-quality control [1], health [2], safety [3], and environmental monitoring [4]. SPR-TFBG sensor usually couples an immobilized bio-specific recognition molecule to the surface of a transducer, which converts a molecular recognition event into a measurable signal, pinpointing the presence of the target molecule [5]. The generated signal is often directly proportional to the target molecule concentration. The concept of combining the recognition properties of biological molecules with the sensitivity of transducers such as optical sensors has led to the emergence of optical SPR-TFBG sensors as valuable sensitive and selective tools in analytical chemistry. Out of all the SPR-TFBG sensors, optical fiber SPR-TFBG sensors have become increasingly popular as an analytical tool in biomedical, biochemical and environmental applications. The main advantage of these sensors, beyond the fact that it is a label-free method, is their ability to give rapid and...
sensitive detection of the target biomolecule in real-time.

TFBG is known for its high sensitivity to change in the surrounding refractive index (SRI) [6]. The formation of a tilted grating structure in the fiber core is responsible for deflecting a fraction of the core mode power and excite a large number of cladding modes in the cladding. Due to the strong mode coupling between the core and cladding modes, a wide band of core-cladding resonance wavelengths is formed in the transmission spectrum of TFBG.

The resonance wavelengths $\lambda_{clad}$ are associated with the grating period, $\Lambda$, effective indices of the core mode, $n_{core}$ and cladding modes, $n_{clad}$ by $\lambda_{clad} = (n_{clad} + n_{core})\Lambda$. When the SRI exceeds the effective indices of the cladding modes, the cladding modes can no longer be effectively confined within the fiber and they become leaky. This explains the decrement in the resonance amplitudes in the shorter wavelength region and it extends further toward the longer wavelength region as the SRI increases. The cut-off resonance wavelength that marks the demarcation between the leaky cladding modes and guided cladding modes is often used as a parameter in the refractometric study for different aqueous and gaseous environment [7]. Furthermore, the dip intensity of the cut-off resonance wavelength is extremely sensitive to small changes in SRI and is proven useful for the detection of biomolecules [8].

The evanescent field of the cladding modes in the TFBG, typically a fraction of the operating wavelength from the fiber surface is the key factor that enables the light-ambient medium interaction. Owing to its limited penetration depth, the evanescent wave can selectively interact with the molecules at the interface, without interference from the free molecules in solution.

The excitation of SPR in the TFBG is a powerful approach to enhance the SRI sensitivity. This is achieved by depositing a thin metal film (e.g. gold and silver) on the fiber glass surface using DC sputtering, vacuum e-beam evaporation, electroless plating method, etc [8]. Unlike the cut-off characteristics of the bare TFBG, the cladding modes couple with the lossy surface plasmon waves at the metal-ambient medium boundary when their effective indices match. This leads to the attenuation of the associated cladding resonance wavelengths within a narrow band region [9-12].

Due to the cylindrical shape of the optical fiber, the thickness of single-sided metal deposition is generally non-uniform in which the metal layer on the side facing the deposition direction is thicker. In contrast, the other half circumference of the fiber remains uncoated. Double-sided deposition by means of manual rotation of the fiber by 180° between two successive depositions is suggested to improve the deposition coverage around the fiber and the SPR performance. Nonetheless, the metal thickness is highly non-uniform in which the minimum thickness on a certain part of the fiber surface can be negligibly small [11]. Rotation of fiber during the deposition process is suggested for forming a highly uniform metal film on fiber surface [13]. However, additional parts are required in the deposition instrument to enable the rotation mechanism. The angle between the tilt plane and the deposition direction is a critical parameter affecting the performance of the SPR-TFBG spectrum [12].

Over the years of development, numerous techniques have been reported for analyzing the optical transmission spectrum of TFBG in sensing applications. Islam et al. have reported, on the shifting of the high-order and low-order cladding resonance wavelength in response to the changes in the SRI [14,15]. Yu et al. [16] have reported the shifts of high-order and medium-order cladding resonance wavelengths for hydrogen gas leakage detection. The spectral analysis based on the shift in SPR wavelength can be used for SRI detection [17]. Due to its high sensitivity, this technique is commonly used for biochemical detection. Caucheteur et al. [18] have demonstrated the detection of small SRI change based on the amplitude shift of the most sensitive cladding mode within the range of SPR wavelength. A similar method had been reported for small biomolecule detection with a detection limit of 0.4 nm [19].

Nevertheless, the optical spectrum of TFBG can be sophisticated, and the analysis can be tedious. Dong et al. have reported the use of integrated transmission power - a simple indicator generated from the TFBG optical spectrum for the detection of ambient humidity [20]. Maguis et al. have demonstrated using Fourier transform to acquire the spatial frequency spectrum of a bare TFBG for signal analysis and detection [21,22].

In this study, we investigate the impact of different deposition orientation with respect to the tilt plane of the TFBG on their output spectrum. Based the double-sided deposition scheme, we study the influence of the deposition angle to the spectral characteristics of the TFBG in terms of cut-off wavelength, SPR bandwidth and attenuation. For the characterization of the sensor as refractometer, the TFBGs are tested using saline solutions with refractive indices in the range of 1.3155 to 1.3620. The optical spectrum is converted into spatial frequency spectrum by using FFT. A frequency parameter can be retrieved from the frequency spectrum and employed as an indicator for in-situ refractometric measurement using saline solutions.

2. Tilt plane orientation and gold deposition

Fig. 1(a) shows the radiation pattern of a bare TFBG excited by a red-light source. It is a ring-shape red light pattern (on the xy-plane) with the TFBG at its center (parallel with the z-axis). The two brightest points on the ring suggest that a large portion of the red-light intensity is scattered from the core to cladding and partly out of the fiber in the yz-plane as indicated by the pink arrows in Fig. 1(b) and (c). The deposition angle $\alpha$ represents the angle between the deposition direction and tilt plane (yz-plane), it dictates the positions of the brightest points of the radiation on the gold coating. Considering the non-uniform profile of the gold coating by single-sided or double-sided deposition schemes, the local coating thickness, $R_{cl}$ where the radiation beam effectively interacts with and the resultant output spectrum, vary with $\alpha$. The relationship between $R_{cl}$ and $\alpha$ is given by

$$R_{cl} = \sqrt{r^2 \sin^2 \alpha + (r \cos \alpha + d)^2} - r, \quad 0 < \alpha < 180^\circ$$

(1)

where $r$ is the radius of the optical fiber and $d$ is the maximum thickness of gold film.

In the fabrication, the TFBGs were written by using a KrF excimer laser and a phase mask with a period of 1081.61 nm. The tilt angle was set at 15° to produce cladding mode resonance wavelengths that span from 1490 nm to 1560 nm and the Bragg wavelength...
was at $\sim 1567$ nm. Prior to the gold deposition process, the tilt plane orientation of each TFBG was identified based on the radiation pattern from the fiber excited by the red-light source. After that, the TFBG was carefully transferred and fixated a metal plate before it was sent for gold deposition process. A batch of gold-coated TFBGs with the same gold thickness, $d = \sim 40$ nm but different deposition angles have been manufactured. Their output spectra are presented in Fig. 2. In the characterisation test, the TFBG specimens were immersed in two different liquid solutions (deionized water, RI $\sim 1.3159$ and 10 wt% saline solution, RI $\sim 1.3332$) to analyse the spectral responses. The RI of the liquid solutions were calibrated using a prism coupler with a 1550 nm light source. It is worth noting that both single-sided and double-sided deposition schemes produce similar spectral properties in TFBG in the study of deposition angle. The related study can be found in reference [12]. However, the double-sided deposition scheme offers stronger SPR properties hence it is adopted for the following tests in this work.

Fig. 2(a) shows the spectra of the TFBG at $\alpha = 0^\circ$. A narrow SPR resonance that separates the wideband of cladding resonances into two regions (see shaded area) can be seen at $\sim 1505$ nm when it is immersed in the DI water and it shifts to $\sim 1514$ nm in a 10 wt% saline solution. At $\alpha = 30^\circ$, SPR resonance is observed at the same wavelength $\sim 1505$ nm in DI water and $\sim 1514$ nm in a 10 wt% saline solution. The major changes in the spectra are the wider SPR resonance bandwidth, more substantial attenuation to the cladding resonance amplitudes within the SPR resonance region and milder attenuation to the other cladding resonance in the longer wavelength region ($> 1505$ nm for RI $\sim 1.3159$, $> \sim 1514$ nm for RI $\sim 1.3332$) as depicted in Fig. 2(b). Similar changes can be observed in the spectra of TFBG at $\alpha = 60^\circ$, as depicted in Fig. 2(c).

The SPR resonance bandwidth expands further and the cladding resonance amplitudes in the longer wavelength region continue to decrease at $\alpha = 70^\circ$ (See Fig. 2(d)). These two regions have been seemingly merged into one at $\alpha = 80^\circ$ and $90^\circ$ as indicated by the shaded areas in Fig. 2(e) and (f). Following the trend of variations in the spectra, it is not difficult to find the connection between the SPR characteristics to the varying RL which are estimated to be 40.0 nm, 34.6 nm, 20 nm, 13.7 nm, 7 nm and $\sim 0$ nm for the spectra in Fig. 2(a)–(f). The lowest value of RL at $\alpha = 90^\circ$ explains why the SPR effect and the attenuation to the cladding resonances in Fig. 2(f) are the weakest. The highest $R_L$ is achieved at $\alpha = 0^\circ$ where the narrowest SPR resonance is attained for this TFBG (Fig. 2(a)). Unlike

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**Fig. 1.** (a) Radiation pattern from a bare TFBG excited with red light laser. (b) Cross section illustration of a TFBG under double-sided deposition scheme. The vertical dotted line indicates the tilt plane of the TFBG. The vertical arrows mark the brightest points of the radiation of the TFBG. (c) Side-view illustration of the tilted grating structure in the fiber. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article).
the other spectra, there is no sign of cut-off attenuation to the cladding resonances in the shorter wavelength region in these spectra because a large portion of the scattered light incidents on the gold-coated fiber surface which contributes to the SPR resonance. In other specimens ($\alpha > 0^\circ$), there is a higher portion of scattered light incidents on the fiber surface that has none or a negligibly thin layer of gold which is responsible for the cut-off attenuation. The cut-off wavelengths of the spectra in Fig. 2(b)–(f), (indicated by the red arrows) are consistent with those found in the bare TFBG as shown in Fig. 2(g). These findings are consistent with the results presented in reference [11].

3. Spatial frequency spectrum and refractometry

The analysis of the TFBG optical spectra can be made easily by transforming them into the frequency spectra by Fourier Transform. The following study presents some examples using some TFBGs at specific deposition angles. Several different parameters and features of the frequency curves at different SRI are extracted and compared. Fig. 3(a) shows the optical spectra of a bare TFBG (uncoated) in a refractometry test using saline solutions. The variation of the cut-off wavelength, $\lambda_{\text{cut-off}}$ with increasing SRI can be well represented by the dotted line with a linear sensitivity of 447.9 nm/RIU. The ‘envelope’ of the guided cladding resonance wavelengths shrinks with increasing SRI.

Fig. 3(b) shows the frequency curves acquired from the Fourier Transformation of a bare TFBG optical spectra. The frequency curves can be seen as the ‘envelop’ of the optical spectra that shrink in bandwidth and the lower frequency edge of the curve at $\sim 0.8 – 1.0$ nm$^{-1}$ represents the $\lambda_{\text{cut-off}}$ red-shifts toward to higher frequency direction with increasing SRI whereas the other edge of the curve is not responding to the SRI. The centroid frequency, $f_{\text{c}}$, offers a linear SRI response is used in the measurement. It is calculated from the centroid of area under the frequency curve in the range of 0.75–1.4 nm$^{-1}$. From the linear curve in Fig. 3(c), the sensitivity is found to be $1.1987$ nm$^{-1}$/RIU.

TFBG coated at $\alpha = 80^\circ$ offers interesting output responses to increasing SRI. The cut-off attenuation in the shorter wavelength region and SPR attenuation that covers a wider band in the longer wavelength region have shaped the optical spectrum into a smaller packet of cladding resonances (Fig. 4(a)). Since both $\lambda_{\text{cut-off}}$ and $\lambda_{\text{SPR}}$ share similar SRI sensitivity, the envelope of the cladding resonances red-shifts but maintains a constant bandwidth with increasing SRI. $\lambda_{\text{max}}$ denotes the wavelength position of cladding resonance with the maximum amplitude. The extraction of this parameter can be easily attained using any data processing software.

![Fig. 2](image-url)
Fig. 3. (a) Optical spectra (b) Frequency spectra of a bare TFBG to different SRI in the range of 1.3159–1.362. (c) Linear relationship between $f_C$ and SRI.

Fig. 4. (a) Optical spectra (b) Frequency spectra of a double-sided coated TFBG ($\alpha = 80^\circ$) to different SRI in the range of 1.3159–1.362. Linear relationship between (c) $f_C$ and SRI.
Similar to the spectra in Fig. 4(b), this TFBG produces a bell-shaped frequency curve but with both of its edges sensitive to SRI change. On the other hand, the centroid frequency of the frequency spectrum in Fig. 4(c) shows a higher RI sensitivity of $1.5784 \text{ nm}^{-1}/\text{RIU}$ if compared with that of Fig. 4(c).

Fig. 5(a) and (b) show the optical and frequency spectra of a gold-coated TFBG ($\alpha = 0^\circ$) at different SRI. The SPR resonance in the optical spectrum can also be observed in the frequency spectrum. A new dip herewith denoted as $f_{\text{SPR}}$ can be detected within the range of $0.9$–$1.1 \text{ nm}^{-1}$ in the frequency spectra. Analogous to $\lambda_{\text{SPR}}$ in the optical spectrum, $f_{\text{SPR}}$ red-shifts with increasing SRI as shown in the inset in Fig. 5(b) and its linear relationship with SRI is presented in Fig. 5(c). In comparison with $f_c$ in Fig. 3 and Fig. 4, $f_{\text{SPR}}$ has an outstanding SRI sensitivity of $4.3336 \text{ nm}^{-1}/\text{RIU}$. On a side note, the lower frequency edge of the frequency curve is unaffected by the SRI, and this is consistent with the earlier observation from the TFBG coated at $\alpha = 0^\circ$ in Fig. 2(a).

Fig. 6 shows the output response of the SPR-TFBG sensor ($\alpha = 0^\circ$) in a saline solution with a varying refractive index. The output spectra are analyzed and recorded by an FBG interrogator (Micron Optics Inc, si155) at the interval of 1 s. The FBG interrogator is controlled by using the MATLAB Application programming interface by Micron Optics Inc (x55 HYPERION MATLAB API v0.9.5.0) in which the FFT algorithm is incorporated in the MATLAB program to process optical spectrum and to determine $f_{\text{SPR}}$ in real time. The average computation time for each spectrum is $\sim 0.168 \text{ ms}$. First, the sensor is immersed in 100 ml of deionized water (H$_2$O) solution and the initial measured value for $f_{\text{SPR}} = 0.8376 \text{ nm}^{-1}$ (Est. RI = 1.3159). Following that, a 10 g of NaCl fine salt is first added into the solution at $t = A$ to raise the salinity from 0 wt% to 9.1 wt%. With the aid of magnetic stirrer in the solution, the dissolution of salt is accelerated and completed within 60 s. During the process, the changes in the frequency spectrum can be observed in real-time in

![Fig. 5. (a) Optical spectra (b) Frequency spectra of a double-sided coated TFBG ($\alpha = 0^\circ$) to different SRI in the range of 1.3159–1.362. The inset shows the shift in SPR frequency, $f_{\text{SPR}}$ at different SRI. (c) Linear relationship between $f_{\text{SPR}}$ and SRI.](image1)

![Fig. 6. In-situ refractometric measurement for a saline solution with varying concentration/refractive index. The secondary vertical axis denotes the estimated refractive index of the saline solution.](image2)
which the \( f_{\text{SPR}} \) rapidly increases from 0.8376 nm\(^{-1}\) and slowly settling down at 0.9001 nm\(^{-1}\). Based on the best-fit linear equation in Fig. 5(c), the estimated RI of the solution is 1.3303. The same exercise is repeated twice, each at \( t = B \) and \( t = C \) to increase the salinity further to 16.7 wt% and 23.1 wt%. The frequency indicator \( f_{\text{SPR}} \) continues to rise to 0.9689 nm\(^{-1}\) and then 1.0251 nm\(^{-1}\). Each transition at \( t = B \) takes about 79 s to complete the salt dissolution, whereas the transition at \( t = C \) takes ~94 s. The estimated RIs at the corresponding steady states are 1.3462 and 1.3592, respectively. Following that, 50 ml of deionized water is poured to the solution at \( t = D \) to dilute the salinity to 16.7 wt%. The signal \( f_{\text{SPR}} \) undershoots for a brief period of time before it settles down at 0.9689 nm\(^{-1}\) (est. RI = 1.3462) which is the same value as the state right before \( t = C \). The transition is almost instantaneous because it takes a much shorter time to achieve the homogeneity of solution. At \( t = E \), an additional 150 ml of deionized water is poured into the saline solution. Similar to the observation at \( t = D \), \( f_{\text{SPR}} \) undershoots for a short period of time before it settles down at 0.9001 nm\(^{-1}\), which is equivalent to the state before \( t = B \) where the salinity is 9.1 wt% and the est. RI = 1.3303.

4. Conclusion

In this work, we have fabricated and experimentally characterized the optical spectra of SPR-TFBGs with different gold deposition angles, \( \alpha \). The excited SPR resonance properties vary with \( \alpha \) due to the non-uniform gold deposition profile and different interaction dynamics with cladding modes in the TFBG. By using FFT, the complex optical spectra of the TFBG can be converted into a spatial frequency spectrum with simpler curve shapes. The frequency spectrum can be seen as a simplified version of the optical spectrum that still inherits the important features such as the cut-off attenuation and SPR resonance from the optical spectrum. Depends on the curve shapes, the centroid frequency, \( f_c \) and SPR frequency, \( f_{\text{SPR}} \) can be retrieved from the frequency spectrum and characterized using saline solutions with different refractive indices. These frequency parameters have shown excellent linear responses to SRI change with an \( R^2 > 0.99 \). The SRI sensitivities of \( f_c \) for bare TFBG and SPR-TFBG at \( \alpha = 80^\circ \) are found to be 1.1987 nm\(^{-1}\)/RIU and 1.5784 nm\(^{-1}\)/RIU respectively. The SPR frequency, \( f_{\text{SPR}} \) of SPR-TFBG at \( \alpha = 0^\circ \) has the highest SRI sensitivity, which is 4.3336 nm\(^{-1}\)/RIU. By exploiting the simple frequency spectral curve characteristics, \( f_{\text{SPR}} \) (the SPR dip frequency) can be easily identified and employed for in-situ refractometric measurement of a saline solution with varying salt concentration. This technology has great potential for various applications such as chemical processing, oil & gas and food & beverage manufacturing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

This work was supported by the University of Malaya, Impact-Oriented Interdisciplinary Research Grant (IIRG) (IIRG028A-2019).

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