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Planar Total Internal Reflection Biofouling Sensors

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Planar Total Internal Reflection Biofouling Sensors

By

Koo Hyun Nam

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requirements for the degree of

Doctor of Philosophy

in

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in the

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of the

University of California at Berkeley

Committee in charge:

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Planar Total Internal Reflection Biofouling Sensors

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by

Koo Hyun Nam
Abstract

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Doctor of Philosophy in Mechanical Engineering
University of California at Berkeley
Professor Liwei Lin, Chair

Planar, integrated microscale sensors utilizing prism-coupler type angular interrogation sensing technique have been demonstrated. The main structure of the sensor consists of an optical prism coupled to a built-in waveguide to introduce Fraunhofer diffraction when light ray comes into the prism from the waveguide. The Fraunhofer diffraction creates spectrum of consecutive rays over the sensing edge of the prism such that there is no need for the bulky scanning mechanisms typically used in other macro scale sensing systems. Two types of sensors are presented: (1) total internal reflection based critical point detection (CPD) sensor, and (2) surface plasmon resonance (SPR) based resonance point detection (RPD) sensor.

The CPD sensor is fabricated by a simple, two-mask process which creates a right angle prism with three sides with lengths of 1, 0.86, and 1.33 mm, respectively in the prototype design and a waveguide with a cross sectional area of 4×0.25 μm². The 0.25 μm-thick core and the 2.5μm-thick cladding layers of the waveguide are made of silicon nitride and silicon dioxide, respectively. The CPD sensing technique measures the shift of the critical point of the total reflection as the results of change of refractive index due to biofouling. Optical simulations are used to validate the working principle and the calculated biofouling sensitivity is comparable to the other optical sensing methods. A baseline measurement has been conducted to verify the operation of the sensor with an error of less than ± 0.002 R.I.U. During a 9-hour biofouling measurement using milk as the media, a change in the refractive index as much as 0.0089 is recorded as the result of biofouling.

The RPD sensing technique employs surface plasmon resonance as its sensing mechanism by measuring the shift of the resonance point with respect to the change of the incident angle. The design and fabrication process is similar to the fundamental structure of CPD sensors with an additional deposition of a thin metal layer on the sensing edge of the prism. The theoretical sensitivity is calculated as 90 deg RIU⁻¹, which is comparable with the state-of-the-art optical sensors at 127 deg RIU⁻¹. The refractive index measurement for selected liquids agrees with the values in the literature with an error range of less than ± 0.002 R.I.U. Furthermore, the refractive index change of biofouling formation is measured to be 0.0078 for a 9-hour experiment using milk as the testing media.
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Most of all, I cannot find words capable of describing my appreciation for my parents. You are the reason for which I live, for why I am the person I am, and for my success. So most of all, I would like to dedicate this doctoral study to you. To my mother, Mrs. Yeon Soon Seo: you are the strongest and sweetest person in the world. And to my father, Mr. Ki Hong Nam: you are my hero, and there is nothing that will ever change that. Mom and dad, you are my tears, smiles, and everything that I can have that is of any value or is worth living for.
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Chapter 1

Introduction

1.1 Prism-coupler Type Angular Interrogation System

Total Internal Reflection (TIR) is a well known physical phenomenon that has been researched and utilized for optical observations for several decades [1, 2, 3, 4, 5]. Used as a sensing technique, it has been appreciated for its ability to provide label-free sensing: that is, analyses without the use of fluorescent or radioactive materials [6, 7]. The presence of the critical points and surface plasmonic evanescent (decaying) waves are two of the most representative mechanisms for sensing employing TIR. Among various TIR applications, the prism-coupler method, also known as attenuated total reflection (ATR) method, is most advantageous [8, 9] as the angular interrogation sensors measure variations with respect to the changes of incident angle of light for high sensitivity and wide range detections [10, 11]. Other techniques such as the optical fiber sensors [12, 13] and the grating-coupler type sensors [14, 15, 16] have been developed, but they are not as versatile as the prism-coupler type sensor in terms of sensitivity and detection range [9].

Conventionally, in the prism-coupler type angular interrogation sensors, the change of an incident light angle is applied by rotating the light source which requires bulky and expensive rotational devices [17]. The rotating light source is especially problematic for micro-scale optical experiments and in-situ online measurements. Another problem arises from the extended scanning time. When used in multi-point measurements, long scanning times may lead to inaccurate results and discrepancies between the sensors. In contrast, waveguide type sensors are typically smaller in size, while their application could be limited because of lower sensitivity and narrower detection range of the propagating light in the guide [8, 18, 19].
Figure 1.1 illustrates the sensitivity versus capacity for miniaturization plot, including waveguide type, intensity measurement type, wavelength interrogation type and angular interrogation type sensors. Clearly, the goal is to make miniaturized sensors with good sensitivity as shown. In this study, we propose to adopt a simple optical phenomenon, Fraunhofer diffraction, to replace the rotating light source employed in the angular interrogation sensing technique for the scanning of the incident light angles within a prism-coupler structure. This principle simplifies the detection mechanism such that the prototype microscale sensor requires only a two-mask fabrication process. This microscale sensor consists of a built-in waveguide with a prism-coupler to take advantage of Fraunhofer diffraction occurring at the end of the waveguide. Since the proposed sensor is fully planar integrated; it can be made using an IC fabrication process. Therefore, integration with other IC devices and micro-size fabrication could be readily available, and a low unit cost should be achievable through mass production. Furthermore, the angular interrogation type sensors typically have better sensitivity and we aim to maintain the sensitivity of the proposed planar total internal reflection sensors.
The proposed sensor could potentially improve the sensitivity and detection range while maintaining the benefits of using a simple waveguide structure as illustrated in Fig. 1.2. There are a wide range of applications that may utilize this sensing structure, including process, motion, gas, and chemical monitoring. Specific characteristics suitable for these applications include: non-destructive nature for material characterization [20]; highly sensitive for in-situ gas sensing [21, 22] and artificial olfactory sensing [23, 24]; and possible benefits from scaling effects which enables the measurement of protein conformation [25], chemical sensing [26], polymer concentration [27], and monolayer formation (biofouling) [28]. This research concentrates on the possible applications for biofouling to reduce the maintenance cost in clean water technologies.

Two types of sensing methods are investigated: Critical Point Detection (CPD) and Resonance Point Detection (RPD), including details in analysis, fabrication, and experiments. Both sensor designs enable: (1) the reduction of sensor size and the elimination of moving parts, (2) the elimination of scanning operation, and (3) planar integration of the entire system. Moreover, small-sized sensors can better enable multi-point local detections for improved sensing accuracy for broader application areas.

### 1.1.1 Critical Point Detection Sensing

The CPD sensor detects the movement of the critical point in order to measure the refractive index changes utilizing the TIR scheme. The shift of critical point in accordance with the change of refractive index of the analyte occurs as is illustrated in Figure 1.3. This sensing method makes use of the rapid drop of intensity at a certain angle of incident light under the TIR. Since this is a simple physical phenomenon, no additional fabrication or manipulation is required for sensing.

![Figure 1.3: Critical point shift on the reflectance profile under TIR conditions with regard to changes in refractive index.](image-url)
When the reflectance change under TIR conditions is combined with the Fraunhofer diffraction by a waveguide structure as proposed, resulting light signal becomes convoluted, as shown in Figure 1.4. Compared to other optical sensors such as the SPR sensor which requires a thin metal layer, the structure materials of this sensor is less vulnerable to erosive wear. Therefore, this type of sensor is much better suited for environment that may require chemical and mechanical polishing on the surface such as biofouling in water desalination stations.

![Figure 1.4: Typical reflectance and diffraction patterns for light of varying incident angles and the convolution pattern of their combination as a resulting sensor signal.](image)

1.1.2 Resonance Point Detection Sensing

RPD sensors measure the refractive index of a material by detecting the movement of resonance points through Surface Plasmon Resonance (SPR), and the shift of resonance point in accordance with the change of the refractive index of an analyte as is illustrated in Figure 1.5. Unlike the CPD sensor, RPD sensors require a thin metal layer to form the metal/dielectric interface where surface plasmons resonate and propagiate. Thin metal coating material and the thickness of the layer are two key factors in the optimization of the SPR signals and gold layer of several tens of nanometers in thickness is commonly used.
Figure 1.5: Resonance point shift on the SPR intensity profile according to changes of refractive index.

When the intensity variations caused by SPR values with respect to the angle of the incident light are combined with the Fraunhofer diffraction, the resulting light signal is a convolution pattern, as shown in Figure 1.6. Compared with other optical sensing methods, this technology could have greater sensitivity. Additionally, the sharply defined output signal offers great clarity for signal detection in practical measurements.

Figure 1.6: Typical SPR intensity profile, diffraction patterns for varying incident angle of light and the convolution patterns as resulting sensor signals.
1.2 Chapter Overviews

Chapter 2 discusses the concept of the proposed prism-coupler type sensing method using Fraunhofer diffraction. A numerical simulation is conducted to investigate the feasibility of this sensor and its sensitivity which is comparable to other current sensing methods. A macroscale experiment using commercial optical components is conducted. The technique is found to be feasible as a refractive index monitoring sensor.

Chapter 3 presents a discussion of the theoretical background of the proposed micro CPD sensing technique for measuring the refractive index of an analyte through the utilization of both Fraunhofer Diffraction and the convolution of reflected and diffracted light. A distinctive feature of the prototype sensor is an integrated waveguide which takes advantage of the Fraunhofer diffraction occurring at the end of the waveguide. The experimental results demonstrate that this sensing technique enables a wider application area than conventional prism-coupler type sensors while maintaining the quality of measurement. Since the material of the sensor structure is stiff to resist erosive wear better than other materials used on the sensing surfaces, tribological theory and techniques are employed to prove the actual superiority of this class of sensor.

Chapter 4 is a study of Surface Plasmon Resonance (SPR) and its application to the CPD sensor as presented in chapter 3. The advantage of SPR technology is its ability, under certain conditions, to measure refractive indices with high selectivity. As is the case with the CPD sensor discussed in Chapter 3, the proposed RPD sensor takes advantage of an integrated waveguide. The numerical simulation and experiments are conducted to investigate the feasibility of this sensor, and the result shows better accuracy than the CPD sensors.

Finally, the chapter 5 concludes the thesis and suggestions for future study are presented.
References


Chapter 2

Macroscale Prism-coupler Type Sensing using Fraunhofer Diffraction

2.1 Introduction

A prism coupler is an instrument widely used to measure the refractive index and thickness of materials as well as to couple light in a waveguide. Prism structure in a prism coupler is utilized to introduce incident light from a variable angle source and to deliver the reflected light from the prism surface to a detector or screen. For most applications, a prism coupler characterizes the properties of an analyte using measurements of reflected light emitted at various angles. Angle scanning devices such as goniometers are usually employed to change the angle of incident light, and the measurement resolution of the prism coupler is dependent on the rotational accuracy of the scanning device. For this reason, prism couplers commonly require a bulky and expensive angle scanning device for high precision measurements.

The prism-coupler method has been researched for adoption in conventional devices such as ellipsometry type sensors [1], and also in very high sensitivity SPR sensing techniques [2, 3, 4]. Among the prism-coupler type SPR sensors, fan-shaped light diverging schemes do not need moving parts (for instance, a goniometer). Thus the latter design has been actively studied in terms of its possibilities in miniaturization [5, 6]. However, due to the limitation that follows from the fact that the divergence of light requires macro scale optical components such as lenses, the prospects are remote for this sensor type being becoming a microscale device.

In this study, the divergence light within a diffraction pattern induced by a simple single-slit is employed in a prism-coupler structure to create a microscale sensor. Therefore, in this chapter, the feasibility of the sensing concept is first verified through macroscale experiments utilizing reliable optical components such as commercial single-slits and right-angle prisms. A precisely constituted macroscale experimental setup generates a TIR response, and a critical
point is detected to analyze the refractive index of an analyte. Microscale fabrication and detailed sensing experiments of CPD sensors are discussed later in Chapter 3. Although SPR is not incorporated into the macroscale experiment in this chapter, the fundamental concept is basically the same. An RPD sensor utilizing SPR is discussed in Chapter 4.

### 2.2 Theoretical Background

#### 2.2.1 Total Internal Reflection

When the incident angle is low with respect to the angle normal to the interface between two media with different refractive indices, a portion of the ray of light striking the interface is transmitted and absorbed, and the remaining portion is partially reflected [7]. However, as the angle of incidence increases, the reflectance of the light increases and eventually reaches a value of 1 (100% reflection) at the critical point (Fig. 2.1 and Eqn. 2.1). The incident angle at critical point is determined by the refractive index of the medium being measured; therefore the critical angle can be calculated to determine the refractive index of a medium to be examined.

\[
R = r^2 = \left( \frac{n_1 \cos(\theta_i) - n_2 \sqrt{1 - \left( \frac{n_1}{n_2} \cos \theta_i \right)^2}}{n_1 \cos(\theta_i) - n_2 \sqrt{1 - \left( \frac{n_1}{n_2} \cos \theta_i \right)^2}} \right)^2
\]  

(2.1)

![Figure 2.1: Reflectance profile and total internal reflection.](image)

The reflectance profiles in Figure 2.1 show how reflectance changes with regard to the refractive indices of three selected media, air, water, and milk. For each medium, there are three critical
points clearly shown on each of the curves. After experimentally determining the position of a critical point, it is possible to redraw the profile, and thus to derive the value of refractive index. This is the basic theoretical background of the sensing methodology employed by the CPD sensing techniques examined in this study.

2.2.2 Fraunhofer Diffraction

Fraunhofer diffraction is a far-field optical phenomenon which can be observed when a wave passes through an obstruction. Typically, light waves passing through slits or holes are diffracted. Fraunhofer diffraction occurs as a result of the interference and reinforcement interactions of incoming and diffracted waves [7, 8, 9]. In general, Fraunhofer diffraction is approximated through the following equation:

\[ I(\theta) = I_0 \left( \frac{\sin \beta}{\beta} \right)^2 \]  

(2.2)

\( \beta \) is called sinc function and is defined as,

\[ \beta = \frac{\pi d}{\lambda} \sin \theta \]  

(2.3)

where \( d \) is the width of the slit and \( \lambda \) is the wavelength of the light source.

![Normalized intensity of light and Fraunhofer diffraction.](image)

Figure 2.2: Normalized intensity of light and Fraunhofer diffraction.

When the light passes through a small opening or slit, the intensity of the spread of the light varies with respect to the angle of diffraction, as shown in Figure 2.2. Due to the reinforcement and destructive interference of light waves, numerous peaks and dark fringes are generated. In particular, the region around the zero-order (central) peak is brighter than the others. This light spreading ability of Fraunhofer diffraction can be utilized to manipulate light waves, especially
in situations that limit the use of optical components such as a lens. In this study, the Fraunhofer diffraction is utilized to enable fabrication of microscale planar devices as well as to minimize the use of complex optical components.

As the distance between the slit and the detector decreases (typically, the position at which the light signal is observed in the CPD sensor), the Fraunhofer diffraction begins to lose its dominance over the final pattern of the light, and the final output light pattern begins to follow Fresnel diffraction [10]. This distance was set to be the minimum design parameter (dimension) of the sensor so that the sensor is only affected by Fraunhofer diffraction. The boundary condition between Fraunhofer and Fresnel diffractions is defined by the Fresnel Number, $F$, which is given by the following equation [11]:

$$F = \frac{d^2}{L\lambda}$$

(2.4)

where $d$ is a width of the slit (the aperture) and $L$ is the distance between the slit and the detector. When the Fresnel Number is much smaller than 1, the Fresnel diffraction begins to dominate.

### 2.2.3 The Convolution of Reflection and Diffraction Patterns

When two or more patterns of light merge, the resulting pattern is a summation of each component, which is called a **convolution**, and the interactions of light waves through reinforcement and interference cause such convolutions. By setting the peak of the zero-order Fraunhofer diffraction at a specific point, the profile of convolution for a light wave can be derived as follows:

$$I(\theta) = I_d(\theta) \otimes R(\theta)$$

$$= I_0 \left[ \sin \left( \frac{\pi d}{\lambda} \right) \right]^2 \cdot \frac{n_1 \cos(\theta_r) - n_2 \sqrt{1 - \left( \frac{n_1}{n_2} \sin(\theta_r) \right)^2}}{n_1 \cos(\theta_r) + n_2 \sqrt{1 - \left( \frac{n_1}{n_2} \sin(\theta_r) \right)^2}}$$

(2.5)

where $\theta_r = \theta + \theta_{\text{crit}} - \theta_{\text{shift}}$

For example, when reflection and diffraction occur simultaneously, the pattern of light at the detector becomes complicated as illustrated schematically in Figure 2.3.
Convoluted by multiple light waves, the resulting pattern is affected by each individual signal. Therefore, careful separation of these signals is necessary if there is a need to study any single pattern contributing to the complex pattern of the convolution. In this study, only the light reflection profile of the reflection-diffraction convolution pattern is allowed to vary, while the diffraction pattern remains fixed. Therefore, image analysis is significantly simplified when compared with the convolution patterns that would result from multiple varying patterns of light.

### 2.2.4 Detection of Biofouling

Biofouling is the consequence of an unwanted accumulation of biological substances on exposed surfaces in aqueous environments. Such surfaces are found in pipeline systems [12, 13], heat sinks [14, 15], marine equipment [16, 17, 18, 19], the blood vessels of the human body, [20] and teeth in the case of dental plaque [21, 22]. The substances that deposit and adhere on these surfaces give rise to thin films of bacteria, algae, and fungi, as well as inorganic material. Left unattended, these biofilms may result in the reduced efficiency of aquatic equipment and the increased frequency of system maintenance. For example, thick biofilms deposited over time result in dramatically increased maintenance costs because the usage of common antimicrobial agents is not usually sufficient to remove the biofilms completely [23]. Today, neither the mechanisms of the biofouling processes nor the procedures needed to control them are clearly understood, such that the monitoring of the growth of biofilms is a fundamental and practical issue.

As biofouling progresses to grow thicker, the optical properties of biofilms change. When the thickness of biofilms increases, their refractive index increases accordingly [24]. However, state of the art biofouling sensors based on optical detection do not take into account
changes in the refractive index of biofilms, and this could be a source of measurement errors in those characterizations [25, 26].

Changes in the refractive index are relatively stable and predictable at the base of the biofilm because its location is away from the disturbances of the fluid medium flowing above. Thus protection is provided by biofouling materials between the base and the biofouling surface. Furthermore, after the initial layer of biofilm is deposited, the newly formed biolayers can have a great variety of compositions as well chemical and physical properties. The initial deposition can be seen as medium or substratum capable of providing opportunities for colonization and competition by other organisms. This unpredictable variation of the biofilm-fluid interface on top of the biofilm surface can affect the layer thickness, diffusion depth of biofoulants, nutrients, and other particulates, coefficient of friction, growth rate of the film itself, tendencies toward the adhesion of organic and inorganic particulates and, significantly, the refractive index. These variables make the properties of the surface layer of a biofilm difficult to predict and, therefore, unsuited to the requirements of contemporary sensing technologies. Consequently, the measurement at the bottom of a biofilm constitutes a more reasonable approach to determining the baseline property of biofilm formations.

For this study, the refractive index at the interface between the surface of a sensor and the biofouling media is measured to study the condition of the biofilm and characterized with respect to time to monitor the formation of biofouling processes.

### 2.3 Numerical Simulation

Numerical simulation allows us to predict the convolution of reflection and diffraction patterns of light and to show the feasibility of the sensing technique proposed for this study. Figure 2.4 shows several cases of convolution patterns with media of differing refractive indices. In the figures below, thick solid lines represent the convolution patterns which are likely to be observed experimentally in measurements of the refractive index. The blue and red dotted lines in the figures illustrate the reflection and diffraction of light, respectively. For each case, the change of reflection pattern is the only factor contributing to the shift of the peak in the convolution curve, and this peak constitutes the signal to be detected.

Unlike the original diffraction and reflection patterns, the peak of the convolution line is sharp, and the tip of this signal clearly shifts with respect to the change of refractive index of the analyte. As depicted in Figure 2.5, the peak shift in the convolution curve is a possible indicator for the measurement of a medium’s refractive index. The result indicates that the changes of refractive index and corresponding critical angle are linearly related; therefore the sensitivity is the slope of the fitting line. The theoretical sensitivity of the CPD sensor used in this simulation was 36 deg. R.I.U.$^{-1}$.
Figure 2.4: Output signals of different refractive indices.

Figure 2.5: Refractive index change versus angle of convolution peak.
2.4 Working Principle

Figure 2.6: Schematic illustration of prism-coupler type sensing mechanism utilizing Fraunhofer diffraction.

Figure 2.6 illustrates the sensing principle of the macroscale experiment setup for a prism-coupler type sensor employing Fraunhofer diffraction. The setup consists of a light source, a single-slit, a right angle prism, and a CCD camera as a detector. The single-slit which is joined to the light source diffracts light, and the reflected light emerging from the prism is captured by the CCD. The divergent light reflects against the interface between the edge of the prism and the analyte, and the refractive index of the analyte determines the position of the critical point in the resulting light pattern. Apart from the utilization of a single-slit, the configuration of experimental setup is identical to that of conventional prism-coupler type sensors [27, 28, 29]. The shift in refractive index of the sensed medium is calculated through the observation of the movement of the critical point positions of the total internal reflection.

The output signals captured by the CCD can record the position of the critical point of the reflected light, and the refractive index of the media is calculated by the derivation based on the Fresnel equation expressed in Equation 2.1 [7]. The path that the light travels is illustrated in Figure 2.6, where \( L \) represents the total distance of light traveling from the input edge of the prism to the CCD, \( D \) gives the distance the light traveling from the reflection point to the CCD, and \( \Delta d \) is the shift of the critical point on the CCD detector. The basic formulation of Snell’s law states:

\[
\theta_{\text{critical point}} = \sin^{-1}\left(\frac{n_2}{n_1}\right)
\]
Therefore, the refractive index of medium being measured is given as follows:

\[ n_{\text{analyte}} = n_{\text{prism}} \sin(\theta_{\text{crit}_i} + \Delta \theta) = n_{\text{prism}} \sin(\theta_{\text{crit}_i} + \Delta d / L) \]  

(2.7)

where \( \theta_{\text{crit}_i} \) and \( \Delta \theta \) are, the critical angle at the initial stage (reference material) and the total angle shift of the critical point due to the change of the refractive index during the experiment, respectively. Likewise, \( n_{\text{analyte}} \) and \( n_{\text{prism}} \) represent the refractive indices of analytes to be measured and the prism, respectively.

2.5 Experiment Protocols and Considerations

2.5.1 Direct Refractive Index Measurement

The critical point of the output signal changes as soon as the prism’s sensing facet (exposed edge) is exposed to medium. However, since total internal reflection is a surface phenomenon, it requires an appropriate period of time for the target medium to wet the sensing surface. Otherwise molecules attaching at the surface will not be distributed homogenously, resulting in an inaccurate measurement. This is a common problem for all optical sensors, and it is an important consideration when there is need for precise measurement.

If the analytes are very viscous and adhesive, it will be difficult to remove the molecules attaching to the sensing surface by purging with the reference material (such as water) when cleaning the apparatus. Therefore, liquids with low viscosity are recommended for earlier testing experiments if several analytes are to be measured sequentially by the same device. The solubility of an analyte in the reference material is also an important factor in deciding the sequence of measurements. Water is used as a reference material in this study because of its good solubility with regard to many other liquids. Viscous liquids such as glycerol are tested in the final stages measurement due to their resistance to the purging process.

2.5.2 Media of Different Optical Properties

In-situ measurements capable of observing the dynamic responses of an analyte are possible with this sensing technique. Examples of dynamic responses include: the mixing of liquids, cell growth and densification, as well as biological film deposition and aging. To observe the formation and aging of biofouling, the sensing surface of the sensor is exposed to the liquid containing the biofoulants which provide both the adhesive molecules necessary for biofouling to occur and the feed nutrients for growth. To monitor and characterize biofouling, the surface refractive index between the prism and analytes is measured and plotted with respect to time.

2.5.3 Sources of Error

The improper arrangement and alignment of a sensor and optical components are common sources of measurement error for most optical experiments. Because the intensity of
light signal at each phase is a main target for observation, a stable and steady light source is required in experiments.

The refractive index is also affected by the temperature of the medium. Therefore, the thermal inhomogeneity, or temperature variation of the analyte and the sensor components through which the light passes could be a source of measurement error. As a result, temperature must be controlled throughout all experiments; otherwise compensation must be added during the data processing process to compensate possible errors coming from temperature variations. Humidity is also a factor that influences the transmission of light, but it is regarded to be a minor source of error.

### 2.5.4 Image Processing

Output signals have been analyzed by Image-pro Plus (Media Cybernetics, USA), an image processing software. The numerical results in Figure 2.7 clearly indicate the detection of a critical point. Here, the critical point is revealed as the position after which the intensity of the output signal rapidly diminishes. The flat region of the curve results from a partially saturated region of the light pattern induced by Fraunhofer diffraction, and after the critical point, the output signal decreases with the drop in reflectance.

![Critical Point](image)

Figure 2.7: Image analysis process illustrating how to find the critical point.

### 2.6 Sensing Experiment

#### 2.6.1 Experimental Setup

Macroscale experiments were conducted using optical components including a commercial single-slit diffraction aperture (precision air slit) and right-angle prism to determine the feasibility of the sensing concept. As shown in Figure 2.8, the setup of this macroscale experiment consists of a 630 nm laser source, a single-slit with a 5 um gap, a right-angle fused silica prism coupled in a container in which several media with different refractive indices for measurement, and a detector (the CCD chip in a webcam).
In the setup, the incident light provided by the laser source is reflected from the interface between the reflecting (sensing) facet of prism and the analyte and exits through the exit edge of the prism. The pattern of reflection is changed in accordance with the refractive indices of the analytes in the medium tank as discussed in previous sections of this chapter. The final optical profile is then captured by the webcam.

### 2.6.2 Experiment Results

Because of the high quality of the commercial optical components used for this macroscale experiment, the signals were clear and the image process generated precise measurements was close to expected values. Figure 2.9 shows the first experiment on a clean water and milk mixture. The experiment started with empty medium tank (air), and water was added and came into contact with the sensing surface of the prism. Milk was then added and a change in the refractive index of the mixture was observed. The figure shows captured signals from each medium as well as the corresponding theoretical patterns of diffracted and reflected light that have been manually separated into discrete patterns. The captured signals are the convolution of the Fraunhofer diffraction and the Fresnel reflection, and the critical point appears on the curve adjacent to the zero-order peak of the diffraction pattern. For the convolution, the diffraction pattern does not move, while critical point shifts along the diffraction pattern according to the change of refractive index of various types of media. These output signals agree well with theoretical derivation given in Equation 2.5.
Figure 2.9: Typical output signal captured by webcam and corresponding theoretical patterns of diffracted and reflected light.

Pattern of Diffraction
Pattern of Reflectance

Typical output signal captured by webcam and corresponding theoretical patterns of diffracted and reflected light.
Observations for the transient behaviors were also conducted to monitor the dynamic responses of biofouling formation. Figure 2.10 shows the typical output signals captured by CCD and its intensity profiles for the milk tested after 2, 4 and 8 hours into experiments, respectively. It is observed that the critical point gradually moved leftwards and the corresponding refractive indices variations can be calculated to quantify biofouling characteristics.

The position of critical point was recorded with respect to time, and the refractive index at each point monitored was calculated as shown in Figure 2.11. As illustrated in Figure 2.6, there were two groups of setups for measurement in this experiment, and each group was defined by a distance, D, of 5cm or 10cm, set between the sensing surface of prism and the detector. Theoretically, the distance should not affect the calculation of the refractive index, and the final refractive index results for both of the cases showed a reasonable tendency and were consistent with theoretical predictions. Although temperature was carefully controlled during experiments, compensation for unexpected temperature variation was conducted and has been displayed as a dashed line in Figure 2.11.
In this chapter, prism-coupler type sensing mechanism utilizing the Fraunhofer diffraction induced by a single-slit is studied to validate the proposed architecture for biofouling sensing. Both numerical simulations and macroscale experiments have been conducted. Fraunhofer diffraction has been generated by using a commercial single-slit, and a portion of the diffracted light has reached the sensing edge of the prism with a broad range of incident angles. The total internal reflection critical point can be determined from the convoluted light with the combined patterns of the reflection and diffraction light patterns. The shift of the critical point in accordance with the change of the refractive index of the analyte has been calculated by numerical simulation. Experimental results from the prototype, macro scale system from several liquids of different refractive indices have shown good consistency with theoretical values. The refractive index of biofouling has been measured continuously with respect to time, and the dynamic responses of biofouling characteristics have been recorded. In conclusion, the macroscale experiment validates the concept of the proposed sensing principle.

Figure 2.11: Position of critical point and corresponding refractive indices with respect to time.

2.7 Summary

In this chapter, prism-coupler type sensing mechanism utilizing the Fraunhofer diffraction induced by a single-slit is studied to validate the proposed architecture for biofouling sensing. Both numerical simulations and macroscale experiments have been conducted. Fraunhofer diffraction has been generated by using a commercial single-slit, and a portion of the diffracted light has reached the sensing edge of the prism with a broad range of incident angles. The total internal reflection critical point can be determined from the convoluted light with the combined patterns of the reflection and diffraction light patterns. The shift of the critical point in accordance with the change of the refractive index of the analyte has been calculated by numerical simulation. Experimental results from the prototype, macro scale system from several liquids of different refractive indices have shown good consistency with theoretical values. The refractive index of biofouling has been measured continuously with respect to time, and the dynamic responses of biofouling characteristics have been recorded. In conclusion, the macroscale experiment validates the concept of the proposed sensing principle.
References

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Chapter 3

Critical-Point-Detection (CPD) Sensing

3.1 Introduction

Total Internal Reflection (TIR) is a well known optical phenomenon when all incoming energy is reflected back to the incident medium [1]. This phenomenon has been exploited in several ways for a variety of sensing techniques and applications. TIR-based measurement techniques have been under investigation for several decades. Notable practical studies began in 1964 with Osterberg and Smith [2] who utilized a prism to couple a light beam into a surface wave. Ulrich and Torge [3] utilized total internal reflection phenomena to characterize the parameters of thin-film. Kitajima and Hieda [4] investigated the use of attenuated total reflection (ATR) to measure the refractive indices of metal-foils. Nee and Bennett [5] studied measurements of the refractive index of transparent materials. And recently, Patskovsky and Meunier [6] developed a phase-sensitive silicon-based total internal reflection sensor using the differential phase shift between p- and s- polarized components of reflected light.

The TIR-based methods have also been studied by many other researchers [7, 8, 9], but they have not been widely utilized because of the development of the Surface Plasmon Resonance (SPR) method which provides higher sensitivity compared with TIR approaches [10, 11, 12]. However there are drawbacks to the SPR technologies. The thin metal layers on the surface of SPR-based sensors are vulnerable to mechanical damage; and this often leads to unreliable measurements. In cases where surface friction could be a concern, the use of TIR-based sensors is more desirable because of their greater structural reliability in resisting mechanical damage including shearing and abrasion.

Complex and large-scale engineered systems could utilize the TIR technology such as pressurized liquid transportation systems in desalination applications [13, 14, 15] and food manufacturing plants [16, 17]. These systems operate on solutions containing solid materials under high pressure. The improvement of the biofouling sensing can assist in the effective
control of the cleaning processes as well as the real-time response to operational faults under severe shearing conditions. Furthermore, there are other demands calling for the elimination of the moving parts in conventional TIR-based sensors, such as the rotation goniometer. Such structural simplification also enables a significant reduction of sensor size and scanning time, especially for multi-points and/or mobile measurements. Furthermore, the planar integration of the TIR sensor reduces the cost in device fabrication.

This chapter investigates the microscale total internal reflection sensing techniques as well as possible applications of the CPD sensor.

### 3.2 Microscale Sensor Design and Fabrication

#### 3.2.1 Sensor Design

![Figure 3.1: Schematic illustration of CPD sensing mechanism.](image-url)

Figure 3.1 illustrates the sensing principle of the prototype sensor which consists of a light source, an integrated waveguide-prism structure, and a detector. The waveguide is designed to deliver light from its open end to the prism-coupler, and the reflected light can exit through the side-facet of the prism. The surface of the prism is exposed to the analytes, and diffraction occurs at the input edge (the interface of the waveguide and prism) to spread light in various directions. This design is similar to conventional prism-coupler type sensors without the need for dynamic scanning of light sources.
Figure 3.2: Schematic diagram of light paths in a prototype CPD sensor.

Figure 3.2 shows the schematic diagram of light paths in a prototype microscale sensor. The waveguide and the materials used for the prism are the two major differences as compared with the macroscale setup presented in chapter 2 while the basic sensing mechanisms are the same. Therefore, the equation to calculate the refractive index of medium being measured is modified as follows:

\[
    n_{\text{analyte}} = n_{\text{nit}} \sin(\theta_{\text{crit, i}} + \Delta \theta) = n_{\text{nit}} \sin(\theta_{\text{crit, i}} + \Delta d / L) \tag{3.1}
\]

where \(n_{\text{nit}}\) is the refractive indices of the silicon nitride which constitutes the core of the prism-coupler. \(\theta_{\text{prism}}\) is a design parameter for the prism selected such that the initial critical point is positioned appropriately on the CCD for ease of observation.

### 3.2.2 Fabrication Procedure

In this study, components of the proposed sensor were constructed separately to simplify fabrication. First the body of sensor which consisted of the prism-coupler and waveguide was built, and the external light source and photo-detector were assembled separately. Coupling and alignment of the device were accomplished after all sensor components had been integrated.

As illustrated in Figure 3.3, a two-mask fabrication process was employed to construct the built-in waveguide with integrated prism. A 2.5 μm-thick silicon dioxide (bottom cladding) layer and 0.25 μm-thick silicon nitride (core layer) were deposited on a silicon substrate (Fig. 3.3a). The body of the prism-coupler and waveguide were defined with the first mask (Fig. 3.3b), and the cross sectional area of the waveguide patterned in this step was 4×0.25 mm². An
additional 2.5 μm-thick layer of oxide (top cladding) was deposited on the top of the structure (Fig. 3.3c). The final structure was defined by the second mask and the use of RIE (reactive ion etching) to create smooth sidewall surfaces and to achieve a good aspect ratio (Fig. 3.3d). Finally, the end of the waveguide was cut-opened by wafer dicing, and an optional polishing was applied to reduce the insertion loss in the light signal. The dimensions of the geometry shown in this section are determined by optical simulations, and this will be discussed in next section.

![Figure 3.3: Micro fabrication procedures for proposed sensors.](image)

Silicon nitride was used as the core layer because its refractive index is higher than that of silicon dioxide to guide light into the device. Standard stoichiometric silicon nitride was deposited using LPCVD (low-pressure chemical vapor deposition), and its refractive index was 2.05.

### 3.2.3 Design Variations

There are fabrication parameters that must be determined for optimal sensor performance. One of the most important parameters is the thickness of each cladding and the thickness of the core of the waveguide, all of which will affect the quality of resultant signal and light intensity.

The thickness of the core layer in the built-in waveguide determines the number of the modes of light the device can accommodate. In order to minimize this number to a single mode of light for a clear resultant signal, the thickness of the silicon nitride core is investigated. In general, the number of modes is inversely proportional to the wavelength of light source and proportional to the diameter of core and the numerical aperture of waveguide. As a reference, number of mode can be simplified as: [18]:

\[
\text{Number of Mode} = \left( \frac{\text{Diat. of core} \times \text{NA} \times \pi \times \lambda}{2} \right)^2
\]

where NA is numerical aperture and λ is the wavelength of the transmission light source. Generally speaking, the strength of signal is directly proportional to the thickness of the silicon
nitride core layer. In other words, the light delivering capacity of the waveguide significantly depends upon the thickness of the core layer. Therefore, the core layer should be sufficiently thick both to maintain optimal signal strength and to compensate for the insertion loss at the entrance of the sensor where the light source is coupled with the waveguide. In this study, the thickness of the core layer was determined by using the thickness that maximizes intensity of the light, which can experience attenuation through insertion and propagation loss, within the geometry of the rectangular waveguide which can allow the single mode of light. Another fabrication constraint is that the stoichiometric silicon nitride cannot be deposited to a thickness of more than 250 nm over silicon dioxide without risking the formation of cracks. To avoid cracking, silicon nitride was deposited to 250 nm in thickness to minimize propagation loss.

The thickness of the cladding layer is another constraint. The cladding needs to be thick enough to prevent excessive propagation leakage of light into the silicon substrate. Figure 3.4 shows several simulation results by using the optical software: BeamPROP (RSoft Inc., USA). The left side of Figure 3.4a is the cross sectional view of the waveguide with simulated light intensity profile and the right side is the schematic three-dimensional illustration of the waveguide with core and cladding layer. Figure 3.4b is the simulations result showing a waveguide with insufficient cladding thickness of 1 μm and the light intensity drops to about 0.025 of the original strength about 1000 μm away from the entrance point of the original intensity and continues to drop quickly. Figure 3.4c shows the same simulation with a 2.5 μm-thick oxide layer and the light intensity is about asymptotic to 0.06 of the original strength at about 1000 μm away from the entrance and afterward. Considering the manufacturing process that thicker cladding layer will be more difficult in the etching process, 2.5 μm is selected as the thickness of the cladding layer.

The sensor was designed to have a prism of 1, 0.86 and 1.33 mm in length, respectively, for the exit, input, and exposed edges (as illustrated in Fig. 3.2). Two larger sensors have also been designed with the same prism angle but larger edges (5 and 10 mm for the input edge, respectively) to characterize the size effects. Table 1 summarizes the important parameters for the prototype device.

Table 1: Summary of Microscale Prism-coupler type Sensor for this study

<table>
<thead>
<tr>
<th>Component</th>
<th>Dimension</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cladding thickness</td>
<td>2.5 μm</td>
</tr>
<tr>
<td>Core thickness</td>
<td>0.25 μm</td>
</tr>
<tr>
<td>Width of waveguide</td>
<td>4 μm</td>
</tr>
<tr>
<td>Input edge (3 sensors)</td>
<td>1, 5, and 10 mm</td>
</tr>
<tr>
<td>PDMS channel height</td>
<td>200 and 300 μm</td>
</tr>
</tbody>
</table>
Figure 3.4: Optical simulation results showing coupling of a single mode of light and the propagation loss during its passage through the waveguide.
3.2.4 Fabrication Results

The quality of sensing results depends on the quality of fabrication processes including mask alignment and the smoothness of the surface. Figures 3.5 and 3.6 show the etched surfaces of the sensing (reflection) and exit edges of the prism and the entrance of the waveguide into the prism-coupler. These figures show the sub-microscale roughness and the poor quality of surface roughness at the entrance to the prism-coupler structure. These are the main sources for the degradation of output light signal. Since the roughness of an etched surface depends strongly on the etcher and etching recipes, using a higher quality silicon dioxide etcher could yield a better surface than the general-purpose etcher that was used for this study. Possible post-processing to smooth the surface could also improve the sensor performance.

![Figure 3.5: SEM images of sensing edge of sensor (x3,800 and x13,000).](image)

![Figure 3.6: SEM pictures of exit edge and waveguide entrance (x10,000 and x370).](image)

In order to reduce light scattering, the exit edge of the prism has been set several tens of micrometers from the edge of the sensor chip. However, as shown in Figure 3.7, damage to the edge of the sensor chip is significant to introduce noises in the output signals.
A completely fabricated microscale sensor is shown in Figure 3.8a. Figures 3.8b and 3.8c show the measured diffraction and reflection patterns of the light traveling through the built-in waveguide and prism-coupler. The waveguide and prism-coupler in the figure were intentionally degraded after deposition processes to make the light path more visible.

### 3.3 Experiments and Results

#### 3.3.1 Experimental Setup

Microscale sensors are relatively difficult to test compared to macroscale sensors because the small dimensions of the waveguide entrance. Reliable but complicated optical components including a laser source and CCD were used in these experiments. As shown in
Figure 3.9, a laser source was directly coupled with the opening of the built-in waveguide of the sensor. The wavelength of the light source used was 780 nm because biofilms, which consist of extracellular polymeric substances (EPS), sediments, and bacteria, are sensitive around this wavelength [19]. Usually, each of the constituents of the biofilm has different optical properties, but the mutual absorbance, which can introduce measurement errors, is relatively small at wavelengths around 780 nm and higher [20, 21]. Furthermore, the reflectance is high when the wavelength of light is greater than about 700 nm such that the output signal of the sensor is stronger [22].

![Figure 3.9: Microscale CPD sensor experimental setup.](image)

As shown in Figure 3.10, a polydimethylsiloxane (PDMS) channel was constructed and placed on the sensor chip and the exposed edge was in contact with the liquid media. To prevent leakage, the size of channel was determined in accordance with the viscosity of the liquid media being measured. For the prototype experiments, a number of analytes including milk were used as media, and the height of the microchannel was set from approximately 200 to 300 μm. The height of the channel diminishes as biofilms grow thicker during experiments; therefore it is important to secure a sufficient initial height to prevent the blockage of the supply of the liquid that contains the biofoulants. To minimize the accumulation of unwanted floating substances as well as to avoid excessive shearing acting of the biofouling formation, the liquid media was introduced at a flow rate at 0.1~0.5 ml/hr.
3.3.2 Calibration

In the case of Fraunhofer diffraction, more than 80% of the light intensity concentrates around the center of the zero-order peak within the range of one eighth of the peak to peak distance as illustrated in Figure 3.11. The prototype sensor was designed to have the critical point of the reference material (water) occur within this range which is selected as a main observation area.

The base angle of the prism, $\theta_{\text{prism}}$, previously illustrated in Figure 3.2, was set to 41°. This angle is close to the incident light angle of the total internal reflection from the silicon nitride to the reference material (water) at 40.56°. The main observation area remains adequate until the refractive index of analyte reaches about 1.4, which corresponds to the incident angle of 43.07°.
Once the sensor was tested with water, ethanol was tested, and was used for calibration purpose. Ethanol is chosen as it can be easily purged or cleaned with water.

### 3.3.3 Direct Refractive Index Measurement

To illustrate the basic sensing principle as a refractive index sensor, direct refractive index measurements were undertaken for a number of liquids, including water, ethanol, acetone, and glycerol. Recorded signals, processed images, and critical points for each of the analytes are displayed in Figure 3.12. Using the imaging software (Image-pro Plus), the positions of the critical points are extracted from the output signals. As illustrated in Figure 3.13, the measured refractive index values were in good agreement with the values in the literature. In this figure the “reference line” shows the divergence of each measurement for numbers reported in the literature. In the case of the glycerol measurement, another sensor with a higher designed prism base angle was used to shift the detection range for the increased refractive index. The error range of the measurements for four selected media with different refractive indices was less than ± 0.002 R.I.U (Refractive Index Unit).
Figure 3.12: Refractive index measurement results for air, water, ethanol and acetone.
Figure 3.13: Experiment results of CPD sensor for different media of known refractive indices.

### 3.3.4 Biofouling Sensing

Figure 3.14 shows three independent sensor outputs in terms of pixel positions (beginnings and ends of output profiles) with respect to time during the 9-hour experiment in the environment of milk and water combination. The downward movements of the TIR critical points (the top symbols) result from biofouling, and the corresponding change in the refractive index is as much as 0.0089. Variations between each individual measurement could be due mainly to the random quality of the biofouling accumulation process as well as fabrication variations of different sensors [23]. The red curve at the bottom of the figure represents the average pixel position of the reference point, the leftmost point of the output signal in the inset picture. The positions of the reference points of individual measurements are indicated by the symbols in the lower part of the figure. As is evident in Figure 3.14, throughout the duration of the experiment, there was very little divergence from the reference point. Since the intensity of the output signal before the critical point is within the region of total internal reflection, the reference point does not shift. Therefore, this observation verifies that the shifts of critical point were due to the change of the refractive index on the sensing surface.


![Figure 3.14: Microscale sensing experiment results of CPD sensor for biofouling formation.](image)

### 3.4 Discussions

#### 3.4.1 General

The experimentation for this research was designed to be simple and straightforward because this study is intended to examine the feasibility of a sensing technique. Therefore, the complicated fabrication process of an integrated light source and photo-detector was not included in the prototype sensor design. A more elaborate and accurate installation of the sensor components would be required for further studies. As a result, it was not possible to obtain exact shape of the output signal from different sensors. However, the general observations as recorded support the feasibility study from the prototype sensors.

During the calibration process, multiple efforts were made to generate the best output signal. First of all, the variables associated with the light source including its power and coupling angle were adjusted to change the quality of output signal. As shown in Figure 3.15, a parallel coupled light source may cause unexpected signal noises induced by scattering against the facets of prism body and by the propagation of light through the silicon dioxide cladding. Therefore, tilting the light source to certain angles provides better signals even though the intensity of light drops due to the reduction of light intensity resulting from insertion and propagation loss.
Secondly, a focusing lens was used to compensate low output signal strength by concentrating the projected light and reducing the spot size of the light. However, manipulation of a light source coupled with a lens was extremely difficult. Finally, the measurement direction of the CCD also affects the quality of the output signal. As shown in Figure 3.16, the output signal that exits the thin silicon nitride core layer is diffracted by the air at the exit edge of the sensor. The lower parts of the light beam hit the silicon substrate, and the reflection of this light can contain noises. Furthermore, when other parts of the light beam are reflected by the rough edge of sensor chip, the noise becomes greater. To avoid these problems and obtain the best image, optimal angles for positioning the detector had to be determined for the individual sensors.

**3.4.2 Tribology**

Wear is the process by which material is removed from a solid surface under mechanical shearing conditions. There are several factors which cause wear, one of which is the impact of particles of solids, liquids, and even gasses against a solid surface [24]. The rate of wear, or the wear volume rate, is defined as:
\[ \dot{V} = k_e \frac{W \cdot s \cdot v^2 \beta}{g \cdot H} \alpha \cdot \beta \cdot \delta \]  

Here, \( k_e = f(v, H, \text{size of particle}) \); \( \alpha \) is the effect of particle speed; \( \beta \) is the angle factor; \( \gamma \) is the effect of material hardness; \( \delta \) is abrasive size effect; \( v \) is particle velocity; and \( g \) is gravitational constant. The above equation shows that wear is inversely proportional to the hardness of material and directly proportional to the abundance of abrasive particles on the surface of interest.

![Particle that causes wear](image)

Figure 3.17: Schematic of erosive wear on (a) a CPD sensor and on (b) an SPR-based (RPD) sensor.

The metal layer is the essential component of SPR-based sensors; therefore once the metal layer is partially eroded or unevenly worn, the sensor loses functionality. As shown in Figure 3.17b, the metal layer used in the SPR sensor, is typically made of gold (Au), which has a hardness of 150 kg/mm² [25, 26]. Consequently, such sensors are vulnerable to erosive wear due to their low hardness value. When exposed to harsh environments, the life expectancy of this type of sensor is shorter than that of CPD sensors, especially when many particles come into contact with the measuring surface. (e.g. pressurized sea water in desalination plants)

However, the CPD sensor shown in Figure 3.17a, which consists of dielectric materials, has a higher hardness value, \( 3.5 \times 10^3 \) kg/mm², than would be found in an SPR-based sensor [27]. Therefore, CPD sensors are less vulnerable to erosive wear.

### 3.5 Summary

The feasibility of utilizing a planar, integrated critical-point-detection type sensor for refractive index measurement and biofouling characterization is demonstrated in this chapter. The CPD sensor for this research presents a novel approach to the usage of Fraunhofer diffraction and introduces the possibility for conventional prism-coupler type sensors to be planar and in the microscale. This study details the working principles of sensing mechanism, design, fabrication and characterizations of the refractive index sensors, including image analysis.
and experimental verifications. The CPD sensor for this study utilizes a simple optical design with a built-in waveguide as a prism-coupler sensor. Furthermore, the fabrication process is simplified by the utilization of only two masks for the entire fabrication process. Optical simulation of this sensor architecture and design/fabrication considerations are discussed. The tribology aspects of the proposed sensor are analyzed. It is predicted this CPD sensor is better suited for harsh environments due to the superior hardness of their exposed surfaces. Several liquids have been tested as base-line characterizations for the sensor, and measurements of refractive indices were in good agreement with the values given by manufacturers (literature) with error ranges of less than ± 0.002 R.I.U. During the biofouling experiments, the sensor measured the change of the surface refractive index of a testing liquid (milk), and a shift of as much as 0.0089 during a 9-hour test due to biofouling has been measured. It is expected that this sensing technique could find many potential applications, especially, where there is a need for in-situ, multi-point local characterization of analytes.
References


Chapter 4

Resonance-Point-Detection (RPD) Sensing

4.1 Introduction

Surface plasmon resonance (SPR) is an optical phenomenon that occurs when plane-polarized light reflects against metal under the condition of total internal reflection. Similar to the CPD sensing technique, this phenomenon may be used for measuring the refractive index of an analyte at the surface of the sensor. SPR has been studied extensively for several decades, especially for chemical and biological experimentations due to its outstanding sensitivity.

Ever since the first investigations of surface plasma waves were conducted by Wood in 1902 [1], innumerable experiments have been conducted, a vast body of research has emerged, and many kinds of optical devices using the phenomenon have been invented. Otto [2] and Kretschmann [3] independently conducted studies on the optical excitation of surface plasmons and developed remarkable experimentations. Pockrand et al. [4] introduced the usage of SPR for the characterization of thin films, while Gordon and Ernst [5] employed the phenomenon to monitor the condition of metal interfaces. Nylander and Liqedberg [6, 7, 8] conducted notable research in several investigations for the possible utilization of SPR for gas detection and biosensing. Biacore International AB, a life science products company, now merged into GE Healthcare, USA [9], opened up the commercial SPR biosensor market in 1990.

There are several different technological approaches to the utilization of the SPR phenomenon. Two of the most representative methods are angular interrogation (the measurement of the change of the resonant angle) and wavelength interrogation (the measurement of the change of the resonant wavelength at a fixed incident angle) [10]. Most devices using either of these approaches are based on the prism-coupler method, and consequently this sensing technology is the most common and widely used in SPR sensing devices. Decladded optical fiber with a thin, metal layer on top of the exposed area is another well-known application using the SPR technique, but its lower sensitivity and narrow detection
range prevent its widespread use.

Despite its exquisite sensitivity, the prism-coupler type SPR sensing technique is not widely used for microscale applications due to its bulky structure. Instead, other kinds of sensors such as the waveguide type are commonly used, although the sensitivity and detection range of this particular sensor are not as good as the prism-coupler method.

This chapter proposes a new sensing technique which enables the microscale prism-coupler type angular interrogation SPR sensing method, named “RPD sensing.” This technology employs the CPD sensor structure discussed in Chapter 3. The potential advantages include possible good sensitivity despite its small device size and the elimination of scanning operation and time. This simple methodology could find a broad range of applicable for a variety of small-scale optical measurements.

4.2 Theoretical Background

Surface Plasmon Resonance

Surface plasmons are electromagnetic waves on the interface shared by a thin metal film and a dielectric material. When excited by photons, these surface plasmons propagate in the direction parallel to such interface. This phenomenon is known as surface plasmon resonance. The existence of surface plasmon resonance can be demonstrated in the simple procedure as illustrated in Figure 4.1. Without the metal layer, a total reflection of light occurs, and 100% of the light is reflected when the incident angle is equal to or greater than the critical angle. When the interface of the prism and the analyte is coated with a thin film of a noble metal (typically gold), the intensity of reflected light suddenly drops and then recovers as the angle of incidence increases. This angle is defined as the Resonance Angle [11].

![Figure 4.1: Schematic of SPR-based RPD sensor.](image)

This phenomenon is a consequence of the presence of surface charge density waves at the dielectric/metal interface and has dielectric constants of different signs [12]. A surface plasma wave (SPW) is an electromagnetic wave, specifically a transverse-magnetic (TM) wave, that propagates along the interface. The SPW is defined with respect to its electromagnetic...
field distribution and its propagation constant, and the propagation constant, $\beta$, is expressed by the following equation:

$$\beta = k \frac{\sqrt{\varepsilon_m \varepsilon_d}}{\varepsilon_m + \varepsilon_d} = k \frac{\varepsilon_m n_d^2}{\varepsilon_m + \varepsilon_d}$$  \hspace{1cm} (4.1)$$

where $k$ is the free space wave number, $\varepsilon_m$ and $\varepsilon_d$ are the dielectric constants of the metal and the dielectric, respectively, and $n_d$ is the refractive index of the dielectric.

When the wave vector of the incident light, $k_p$, matches the wave vector of the surface charge density waves, $k_{sp}$, the surface plasmons resonate, and the electromagnetic field of the SPW is confined at the interface and decays evanescently [13]. This resonance point is usually displayed as an upside-down peak in the intensity profile of the detector’s output signal. The change in the refractive index gives the change of the propagation constant of the SPW propagating along the metal layer, and this can be detected by monitoring the intensity profile of output signal [14].

There are several types of sensors to detect the resonance point, and the sensor design for this study is based on the angular interrogation technique which measures optical signals with regard to the change of the incident angle. This sensing technology is acknowledged to offer a higher sensitivity better than other kinds of SPR sensors, and it will be discussed in Section 4.7.2.

### 4.3 Numerical Simulation

A numerical simulation was conducted to observe the output signal of this sensing technique, and the light patterns that resulted from the convolution of the diffractions and reflections of the light affected by SPR were plotted. Figure 4.2 shows the result of a numerical simulation of analytes (e.g. water containing biofoulants) with different refractive indices. The solid lines represent convolution patterns, dotted blue lines illustrate the SPR signal, and dotted red lines illustrate the diffraction of light. As depicted in these graphs, the peak of the convolution line is sharp, and the tip of the signal’s upside-down peak clearly shifts to the right with respect to the changing refractive indices of the analytes.

Using the resonance angles corresponding to the peak’s shifting positions, the refractive index of the analyte was determined and graphed against the incident angles as shown in the Figure 4.3. The sensitivity of the SPR sensor used in this simulation was acquired from the slope of the fitting curve to be 90 deg. R.I.U.$^{-1}$.

Figure 4.2: Results of numerical simulation with different refractive indices.

Figure 4.3: Refractive index change versus the angles of the convolution peaks.
4.4 Working Principle and Design

The basic structure of the RPD sensor used in this study is the same as the CPD sensor discussed in the preceding chapter. As shown in Figure 4.4a, it differs only in the theory of its operation and in the shape of the output signal generated. There are several types of SPR sensors, and one of the most common setups, shown in Figure 4.1, is the Kretschmann configuration. This type of SPR sensor was chosen for present study because of its prism-coupler type is equivalent to that of a CPD sensor, and, consequently, such a configuration requires no structural modification of the CPD sensor setup utilized in Chapter 3.

![Figure 4.4](image)

Figure 4.4: (a) Schematic diagram illustrating RPD sensing mechanism; (b) surface plasma wave (SPW) formation within metal, biofilm, and background.

In cases where the target analyte or biofouling formation is as thick as the penetration depth of the SPW (usually several hundreds of nanometers) [14], as shown in Figure 4.4b, the real part of the propagation constant, $\Delta\beta$, which is explained in Section 4.2.1, is changed by the refractive index change of the analytes. The relationship between the propagation constant and the refractive index change is approximately defined as follows:

$$\text{Re}\{\Delta\beta\} \approx k\Delta n$$  \hspace{1cm} (4.2)

where $k$ is the free-space wave number discussed in the previous section in this chapter.

When a light is reflected at the boundary of the metal layer and the prism-coupler, the evanescent wave propagates along the interface with a constant determined by the incident angle of light. Therefore, the measured incident angle of the light at the event of resonance (the resonance point or upside-down peak in Figure 4.2) can give the propagation constant, and, therefore, the change in the refractive index of the analytes.
4.5 Microscale Sensor Fabrication

The basic structure of the RPD sensor used this study is the same as the CPD sensor discussed in the previous chapter. In other words, the structure of the CPD sensor is adopted for the construction of the RPD sensor. There is only a single additional step required, the deposition of a layer of metal on top of the sensing (exposed) edge of the CPD sensor.

Figure 4.5 shows the additional fabrication step required for the construction of the RPD sensor. As shown, photoresist was used to specify the area to be metalized through a lift-off process. There are two ways to metallize the sensor, but for easy deposition setup, a metallization of only the reflection edge was conducted and 47 nm of gold (Au) was deposited with an e-beam evaporator along with a 3 nm titanium adhesion layer. The thickness of the gold layer was determined by a numerical simulation that will be discussed in Section 4.7.1.

The quality of the metal layer affects the quality of the output signal because the roughness of the prism’s reflection edge is critical for this microscale sensor. Figure 4.6 shows the quality of gold layer deposited on the reflection (sensing) edge of the sensor. As in the previously fabricated CPD sensor, a sub-microscale roughness has been induced during the etching process and this would affect the quality of output signal.

Figure 4.5: Two methods of metalizing the SPR sensor.
4.6 Experiments and Results

4.6.1 Experimental Setup

Measurements using an RPD sensor were conducted with a setup that was basically identical to the one discussed in the preceding chapter. This sensor consisted of the optical components used in the CPD sensor, and the experimental protocols under which the experiments were performed were the same. However, since the surface plasma wave (SPW) used to generate the resonance is a transverse-magnetic (TM) wave, a half-wave plate was added to the experiment setup to filter light from the laser source in order to achieve an appropriate polarization state of that light (Figure 4.7).
4.6.2 Image Processing

The profiles of the measured output signals of an RPD sensor differ from those generated by a CPD sensor, although the way to find the resonance point (the inverted peak) is identical. Figure 4.8 shows numerical results that clearly indicate the detection of the resonance point which is apparent as the position at which the intensity of the output signal dips suddenly. The flatness of the curve from about 180 pixels to around 300 pixels is the result of a partially saturated region of the light pattern induced by Fraunhofer diffraction, and the intensity profile in the region after the resonance point shows effect of the convolution of the diffracted light and the SPR.

![Image analysis process illustrating how to find the resonance point.](image)

4.6.3 Effect of Polarization

General-purpose polarizers do not prove sufficiently precise performance. Therefore, for accurate measurement, calibration of polarizer is necessary. Figure 4.9 shows changes in the output signal and the corresponding polarization state of the light wave. In the figure, the polarizer was set to find the output signal with the sharpest resonance point as a means of calibration.
Figure 4.9: Image analysis process illustrating the resonance points of different polarization states of light.

### 4.6.4 Experimental Results

As was the case with the CPD sensing technique studied in Chapter 3, four different media were selected and tested. Figure 4.10 shows the measurement of the resonance point of each medium for this test rather than the critical point as was the case in the earlier examination of CPD sensing. Figure 4.11 compares the refractive indices obtained from the experimental results with values in the literature.

Based on its theoretical performance, the RPD sensor was expected to display better selectivity. However, the actual result shows measurement errors in the range of ± 0.002 R.I.U. These errors are independent of the SPR-based sensing method and arise from problems with the alignment of the optical measurement set up as well as fabrication defects and problems in the resolution of the measurement devices. As discussed in the preceding chapter, the alignment of the optical devices is among the factors most likely to contribute to error. The incident light that scatters on the side of the exit edge due to its roughness creates additional difficulties for precise measurement.
Figure 4.10: Refractive index measurement results for water, ethanol, and acetone.

Figure 4.11: Experiment results of RPD sensor for media of known refractive indices.

The procedure employed in the CPD sensor test for biofouling discussed in Chapter 3 was utilized in this test. As shown in Figure 4.12, during the 9-hour experiment three
independent sensor outputs were measured in terms of pixel positions with respect to time. The downward movements of resonance points (the symbols associated with the blue curve above the inset) are results of biofouling. The corresponding refractive index change was 0.0078, a value similar to the test results for the CPD sensor, and as was the case in the previous test, the reference point did not shift significantly.

Figure 4.12: Microscale sensing experimental result of the RPD sensor test for biofouling formation.

4.7 Discussion

4.7.1 Design Variations

In addition to the design variations for the CPD sensor discussed in the preceding chapter, the optimal thickness of the additional metal layer requires investigations. Figure 4.13 depicts the simulation results detailing signal response as a result of SPR based on Equation 4.1 and 4.2. As shown in the figure, 47 nm was found to be the theoretical optimal value for the thickness of the gold layer [15, 16]. Deep peaks ensure clear detection of resonance point, and the upside-down peaks (dips in reflectance) at the resonance point observed for other thickness values were shallower. The actual output signals measured in this study showed somewhat shallow resonance points. The major cause for this problem was found to be thicker depositions of gold layer induced by a non-uniform deposition process of evaporator used during fabrication.
4.7.2 Determination of Sensitivity

The most attractive element of the SPR sensor is its high sensitivity for a wide range of scientific measurements. Homola et al. conducted notable and practical studies on the sensitivity of several types of SPR sensors [17, 18]. In their papers, they began with basic equations, such as Equation 4.1, describing the physical phenomena to mathematically derive theoretical sensitivities for each case. The theoretically derived sensitivity of prism-coupler type angular interrogation was determined to be 127 deg. R.I.U. \(^{-1}\), while other types of SPR sensing structures such as wavelength interrogation and intensity measurement with prism or grating coupler-based SPR sensors had relatively lower sensitivities. Under the conditions for the usage of a goniometer with an angular resolution of \(1 \times 10^{-4}\) deg. [7], the resolution was calculated to be \(8 \times 10^{-7}\) R.I.U. as expressed in following equation:

\[
R_{\text{measurement}} = \frac{CR_{\text{component}}}{S_{\text{sensor}}} \quad (4.3)
\]

where \(R_{\text{measurement}}\) is the resolution of the measurement, and \(CR_{\text{component}}\) and \(S_{\text{sensor}}\) are the resolution of the component such as a goniometer, a wavelength modulator or a detector, and the sensitivity of the sensor, respectively.

For this study presented in this chapter, the theoretical sensitivity of the sensing technique was numerically derived by simulations, as discussed in Section 4.3. As mentioned previously, the sensing device utilized here is a prism-coupler type angular interrogation SPR-based sensor. The sensitivity acquired by the simulation was 90 deg. R.I.U. \(^{-1}\), which compares reasonably well to the mathematically derived value from the Homola’s work. However, unlike the Homola’s calculation, since angle scanning is replaced by Fraunhofer diffraction, there is no angular resolution for this work. Therefore the resolution of the measurement instrument is the only factor which determines the resolution of the sensing system. Therefore, a resolution of
the detector is required to be considered in the resolution calculation as it is able to deliver the best performance to the system. One example of detector resolution is setting a CCD of 5 um-pixel size placed 5cm apart from the sensor, and this gives approximately $1 \times 10^{-4}$ deg. of angular resolution, which is equivalent to Homola's calculations. For this simulation model, the resolution was calculated to be $1 \times 10^{-6}$ R.I.U using Equation 4.3. As expected, the sensitivity of SPR-based model is comparable to the conventional prism-coupler type angular interrogation SPR sensor, which has the highest value reported, and it is higher than that of the CPD models discussed in Section 2.3. Table 2 summarizes the values for the sensitivity and resolution discussed in this section.

**Table 2: Summary of sensitivity study for various sensing techniques.**

<table>
<thead>
<tr>
<th></th>
<th>Sensitivity/Component Resolution ([RIU⁻¹])</th>
<th>Resolution (RIU)</th>
<th>Reference</th>
</tr>
</thead>
<tbody>
<tr>
<td>SPR-based sensor</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Angular interrogation</td>
<td>1.27×10⁶</td>
<td>8×10⁷</td>
<td>[7, 17]</td>
</tr>
<tr>
<td>Wavelength interrogation</td>
<td>4.85×10⁵</td>
<td>2×10⁶</td>
<td>[17]</td>
</tr>
<tr>
<td>SPR-based sensor</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Intensity measurement</td>
<td>5.75×10⁴</td>
<td>2×10⁵</td>
<td>[17, 19]</td>
</tr>
<tr>
<td>SPR-based sensor</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Angular interrogation</td>
<td>4.03×10⁵</td>
<td>2×10⁶</td>
<td>[7, 17]</td>
</tr>
<tr>
<td>SPR-based sensor</td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>Waveguide type</td>
<td>2×10⁴</td>
<td>5×10⁵</td>
<td>[17, 20]</td>
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<td>RPD sensor (SPR-based)</td>
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<tr>
<td>Angular interrogation</td>
<td>9.0×10⁵</td>
<td>1×10⁶</td>
<td>Section 4.3</td>
</tr>
<tr>
<td>CPD sensor</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Angular interrogation</td>
<td>3.6×10⁵</td>
<td>3×10⁶</td>
<td>Section 2.3</td>
</tr>
</tbody>
</table>

## 4.8 Summary

A prism-coupler type angular interrogation sensor was selected to demonstrate the SPR devices and modified to remove the bulky rotating light source. This work has led to significant benefits in size reduction to facilitate possible multi-point measurements. In order to verify the feasibility and to calculate the theoretical sensitivity of the sensor, a numerical simulation was conducted. The result indicates that the sensor developed in this study has sensitivity of 90 deg. R.I.U⁻¹. The body of this sensor is based on the CPD sensor in chapter 3.
and the only modification is the deposition of a thin metal layer on the sensing edge at the interface between the sensor and the analyte. Once the prototype sensor was fabricated, experiments were conducted with a setup very similar to the one used previously for the prototype CPD sensor. The experimental results were collected through image processing and the use of a polarizer. These results were similar to those of the CPD sensing technique.
References


Chapter 5

Conclusions and Future Work

5.1 Conclusions

The feasibility of prism-coupler type angular interrogation sensing using a planar, integrated microscale sensor with a built-in waveguide has been investigated with two kinds of sensing mechanisms, the CPD and RPD sensors. The feasibility of the concept of this sensing technique was verified through macroscale optical experiments as discussed in Chapter 2. The microscale sensors, on the other hand, have been designed to be as small as a few hundred of micrometers, and these small structures are based on the utilization of Fraunhofer diffraction which is observed at the end of the built-in waveguide. This diffraction is appropriately positioned and firmly aligned within the sensor, and the built-in waveguide successfully generates the convolution pattern of the combined diffraction and reflection at the detector obviating the necessity of a scanner and, therefore, eliminating the time required for scanning.

As explained in Chapter 3, the prism-coupler type of angular interrogation sensing was applied to a micro-sensor, classified as a CPD sensor. This CPD sensor has been designed and successfully created through a simple two-mask fabrication process. The results of optical simulations demonstrate the conceptual working principle of the sensing technique, and provide a theoretical sensitivity comparable to that of other types of optical sensing methods. Successful measurement has been conducted, and the results have been substituted into the governing equations to determine the refractive index of the analytes. During the measurements of several liquids, the error range has been evaluated at less than ± 0.002 R.I.U. Although the error range of this prototype sensor is quite large compared to the theoretical sensitivity, the measurement results are in good agreement with the reported values. A testing liquid (milk) has been employed to monitor biofouling as a medium of changing refractive index. The change measured was as much as 0.0089 R.I.U. during a 9-hour experimental duration. This dielectric optical sensor could find applications in monitoring systems which measure biofouling under conditions of erosive wearing.
As has been established in Chapter 4, the prism-coupler type of angular interrogation sensing method can be applied to a dielectric micro-sensor with a metal layer on top, classified as an RPD sensor. On the basis of the CPD sensor structure previously studied, an RPD sensor was designed and fabricated. Optical simulations were conducted prior to the actual experiments in order to predict the optical responses of the output signals (the pattern of the convoluted light) as a reference as well as to verify the feasibility of this sensing scheme. The theoretical sensitivity of this proposed RPD sensing technique has been evaluated, and the sensitivity of the technique has been determined to be as good as that of the state of the art sensing method (the goniometer-driven prism-coupler type angular interrogation sensing method). Several common liquids with different refractive indices have been tested with the fabricated RPD sensor, and the refractive indices measured are in agreement with the reported values within an error range of less than ± 0.002 R.I.U. For biofouling formation characterization, the average refractive index change has been measured as 0.0078 R.I.U. during a 9-hour experiment. It has been determined that the error range of the sensor is similar to that of a CPD sensor. However, because this error is mainly the result of a low quality of manufacturing process, the problem could be solved by refining the sensor-fabrication technique. In summary, the sensitivity of the RPD sensor could as good as the bulky conventional SPR sensor, while additional advantages are introduced. The significant reduction of device size and the elimination of scanning time by this RPD sensor technology could lead to a wide spectrum of applications, especially in multi-point sensing and mobile sensing devices.

5.2 Future Directions

Throughout this research, the theoretical bases and feasibility of microscale prism-coupler type angular interrogation sensing techniques utilizing simple prototype sensors have been examined in order to provide preliminary foundations for future study. In this section, a variety of advanced applications and the methods of their fabrication as well as advanced sensing techniques are discussed.

5.2.1 Self Cleaning Sensor using UV and F-IR Ray

Ultraviolet radiation (UV) is known to kill micro-organisms including bacteria [1]. Since the majority of biofouling processes are the results of microorganisms, UV rays can be used to eliminate the chief cause of this problem [2]. Likewise, the thermal energy of Far-Infrared (F-IR) radiation could breakdown fouling [3, 4]. When these two forms of radiation are applied consecutively in a localized area, a significant reduction in the biofouling within that area can be achieved. Using the UV and F-IR rays in the CPD sensor, the accumulated biofouling on the surface of the sensor could be removed; hence the sensor could be reset to its initial condition without the use of removal agents which are often very difficult to apply non-intrusively. As illustrated in Figure 5.1, the provision of those rays from the exit edge of the prism will avoid the intensity reduction caused by the waveguide on the input edge of the prism.
Figure 5.1: Schematic illustrating a strategy to remove biofilm deposition from a sensor using ultraviolet and far-infrared rays as a means of cleaning.

5.2.2 Integration of Laser Diode and Photo Detector

As discussed in the earlier chapters of this study, the construction of the prototype sensor was simplified; therefore both the light source and the detector were excluded from integration into the device during fabrication. To construct complete sensor device unit capable of working independently, a laser diode [5, 6, 7] and photo-detector [8, 9] must be integrated into the sensor chip along with the prism-coupler and built-in waveguide. This complete sensor device is expected to be used directly for multi-point detection for characterization of inhomogeneous materials in large-scale plants.

The integration of an energy harvester and RF transmitter into a sensor is also possible for a stand-alone, self-powered device. A possible example is a prism-coupler type angular interrogation sensor driven by a piezoelectric generator, which emits wireless signals used to characterize an analyte [10]. For example, in water systems, small water-powered sensors embedded at multiple points may be employed for monitoring the system (Figure 5.2).

Figure 5.2: Schematic of a self-powered sensing device.
5.2.3 Measurement of phase difference between s- and p-polarizations

Along with measurement of light intensity, another method for determining the refractive index of an analyte is offered by the measurement of the phase difference, also induced by total internal reflection, between s and p polarizations [11]. According to the Fresnel equation, reflectance, in fact, incorporates two components: the reflectance of the light polarized with respect to the direction of propagation of electric field (TE, s-polarized); and the reflectance of light polarized with respect to the magnetic field (TM, p-polarized). The reflectance given in terms of these discrete values is usually denoted as $R_s$ and $R_p$, respectively, and given by [12]:

$$
R_s = \frac{n_1 \cos \theta_i - n_2 \sqrt{1 - \left(\frac{n_1 \sin \theta_i}{n_2}\right)^2}}{n_1 \cos \theta_i + n_2 \sqrt{1 - \left(\frac{n_1 \sin \theta_i}{n_2}\right)^2}}, \quad \text{and} \quad R_p = \frac{n_1 \sqrt{1 - \left(\frac{n_1 \sin \theta_i}{n_2}\right)^2} - n_2 \cos \theta_i}{n_1 \sqrt{1 - \left(\frac{n_1 \sin \theta_i}{n_2}\right)^2} + n_2 \cos \theta_i}
$$

(5.1)

To measure the refractive index of an analyte, the phase difference between the s and p-polarizations of the reflected light is measured by using light sources of differing polarization states. In such a case, the refractive index can be obtained by substituting the measurement values into the Fresnel equation. For the sensor used in this study, the comparison of two output signals obtained at different polarization states can provide information to calculate the change in refractive index for an analyte.

![Figure 5.3: Response of reflectance in accordance with incident angle for s- and p-polarized light.](image-url)
5.2.4 Measurement in Total Internal Reflection Region

The cloudiness of suspended particles in a liquid is referred to as turbidity and is typically measured through the absorption and scattering of light by the suspended solids. This sensing method has been traditionally used as a means of monitoring biofilm development [13, 14]. However, the technique is accurate only when the particles suspended in the liquid medium are homogeneously distributed — an event which has limited the ability of this sensing method to achieve reliable measurements.

Despite its unsuitableness for accurate measurements of biofouling, the turbidity measurement is very simple and efficient. The region in the total internal reflection on the reflectance profile does not change until the critical point appears. In other words, the region after critical point conserves the original pattern of its diffracted light. Therefore, if there is any change in the region, it must be due to a change in turbidity between measurements. This turbidity measurement could be used as a secondary measuring tool for more precise characterization of analytes.

5.2.5 Digitated Sensor Geometry

![Detector 1, Detector 2, Detector 3](image)

Figure 5.4: Schematic of Three Exit Waveguide sensors and their corresponding points of measurement on a convoluted light profile.

As the output signal exits the prism, there is a great possibility that the signal will become weaker or distorted by the external environment between the detector and the exit edge of the prism. The solution to this problem is to build exit waveguides. As shown in Figure 5.4, three separate exit waveguides are illustrated. Each guide picks up the signals corresponding to a fixed reflectance angle and only three points appear on the graph as an example. The exit waveguide encases the output signal up to the detector to prevent weakening or distortion of the signal [15]. Because only guided signals reach the photo-detector, there should be less noises resulting from light scattered at the prism’s exit edge or from outside light.
Furthermore, there is a good chance that the scattered light at the reflection edge inside the prism will not fall within the insertion range of the exit waveguides. Therefore, the waveguide itself could have a filtering capacity. As discussed earlier in this section, it is possible to integrate the laser diode and photo-detector into the design and it would be straightforward to add the additional waveguides.

5.2.6 Thickness measurement of SPR-based sensor

During the discussion of the working principle in Section 4.4 (Equation 4.2), only the case of an analyte or biofilm of thickness comparable to or thicker than the depth of the SPW field was considered. However, if the thickness of medium to be sensed is much thinner than the SPW field depth, the propagation constant is expressed as follows:

\[ \text{Re}\{\Delta \beta\} = \frac{2n_r n_s k^2 d}{\text{Re}\{\varepsilon_m\}} \Delta n \]  

(5.2)

where \( n_r \) is the refractive index of the reference material [16]. This suggests that it may be possible to discover a way to measure a very small thickness change in a biofouling formation at the initiation stage of tens of nanometers if the refractive index of the biofilm is provided by another measuring device such as, for example, a CPD sensor [17].

5.2.7 Sensor using Fresnel diffraction

Theoretically, the smallest dimension of the sensor design used in this study is about 300 μm, the minimum scale at which Fraunhoffer diffraction becomes dominant. If the distance of light travel is less than 300 μm for this setup, Fresnel diffraction is more significant than Fraunhoffer diffraction. In such a case, the sensor design must be modified. In the event of light waves incident upon a diffraction grating, the resultant image as illustrated in Figure 5.5b (Talbot image) can be acquired when the detector is set at the proper distance from the grating plane [18, 19]. Figure 5.5a shows the conceptual design of the sensor using Fresnel diffraction with all aforementioned factors considered.

Figure 5.5: (a) Schematic of conceptual design of sensor using Fresnel diffraction, (b) corresponding intensity profile.
In the figure, the prism has edges of multiple angles of decline, and each of the angled edges corresponds to an individual slit of the diffraction grating. Each ray propagating through the individual slits is reflected by the angled edge of prism, and the intensity of this reflected light will indicate the position of the critical point. The governing equation for this principle is derived as follows:

\[ I(\theta) = I_s(x(\theta), z(\theta)) \otimes R(\theta) \]

\[ = I_0 \frac{1}{4} \left[ 1 + 2m \cos \left( \frac{\pi x}{L} \right) \cos \left( \frac{2\pi}{L} \right) + m^2 \cos^2 \left( \frac{2\pi}{L} \right) \right] \frac{n_1 \cos(\theta_i) - n_2 \sqrt{1 - \left( \frac{n_1}{n_2} \sin(\theta_i) \right)^2}}{n_1 \cos(\theta_i) + n_2 \sqrt{1 - \left( \frac{n_1}{n_2} \sin(\theta_i) \right)^2}} \]

(5.3)

where the factors in first bracket specify the influence of Fresnel diffraction, and the values in the second bracket refer to the influence of the reflected light.
References


