Application of Fiber Optic Surface Plasmon Resonance Sensor for Measuring Liquid Refractive Index

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ABSTRACT: An optical fiber sensor based on surface plasmon resonance (SPR) phenomenon that can be used for liquid refractive index testing is presented in this article. The effect of various parameters like the refractive index of the core and the liquid type on the shape of the SPR spectrum is analyzed, utilizing relative spectrum measurement technology. The relationship curves between several kinds of liquids with the same refractive index with their resonance wavelengths are also obtained using the sensing probe. Furthermore, experimental results of liquids with high refractive index reveal that the improved probe with SiO film has the ability to extend the detection upper limit of refractive index and keep the toughness of optical fiber at the same time.

Key Words: surface plasmon resonance, optical fiber sensor, refractive index, SiO film

INTRODUCTION

SURFACE plasmon resonance (SPR) is a quantum optical-electrical phenomenon which arises from the interaction between light and a metal surface. It is highly sensitive and precise to the change of refractive index of the environmental medium on the surface of the thin metal film (Miwa and Arakawa, 1996; Homola and Sinclair, 1999). Over the past decade, the research on the sensor based on the SPR effect has achieved great development in the fields such as immune distinctive recognition, film-layer character analysis, and detection of DNA as well (Fontana et al., 1990; Jorgenson and Yee, 1993; Weiss et al., 1996; Slavik et al., 2001). This optical fiber sensor based on the SPR effect, which represents the highest degree of miniaturization, was first developed in Jorgenson et al. (1993). It is a novel biochemical sensor that combines the optical fiber technology and the SPR effect.

Research on the influences of the key parameters of SPR probe, liquid concentration as well as type on the shift of resonance wavelength, will provide more basic information for the research and application in the field of biomedicine, environmental surveillance, and remote sensing (Alonso et al., 1993; Diez et al., 2001). Therefore, several different kinds of chemical liquids with the same refractive index are measured in this study.

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This sort of sensor generally uses conventional quartz optical fiber with a refractive index of 1.468 as the basic material. Its detection range for liquid refractive index must be lower than 1.468 due to the characteristic of the SPR effect. If we employ a special optical fiber with a high refractive index to enlarge the measurement range, the toughness of the optical fiber will be considerably reduced and the cost will be increased. These disadvantages will limit the application of this sensor. For solving these problems, the structure of the SPR probe is also redesigned. And the experimental results obtained are in agreement with the theoretical analysis.

SENSOR STRUCTURE AND PRINCIPLE OF OPERATION

SPR Excited on a Multimode Optical Fiber

Figure 1 shows the structure of an optical fiber SPR sensor. When a linearly broadband polarized light is coupled into an optical fiber, it will produce dispersion and form many monochromatic p lights, which transmit at different total reflection angles θ_i. According to the complex dielectric of metal, the incidence lights will partly propagate into the interior of the silver layer and form the evanescent wave at the interface between the core of the fiber and the silver film. At most wavelengths, the metal acts as a mirror, reflecting virtually all the incidence light. Under certain conditions, the vector of light waves k_x match with those of surface plasmon waves k_{sp}, which are charge density

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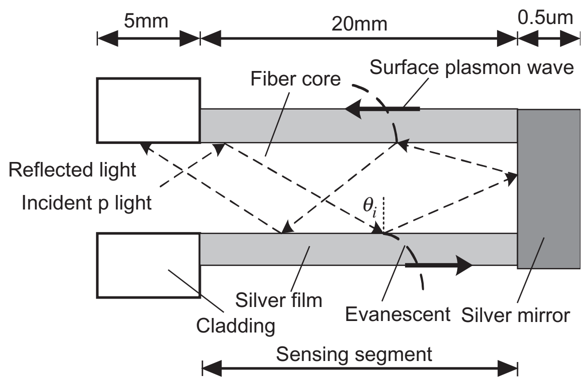

Figure 1. *Structure of SPR optical fiber probe.*

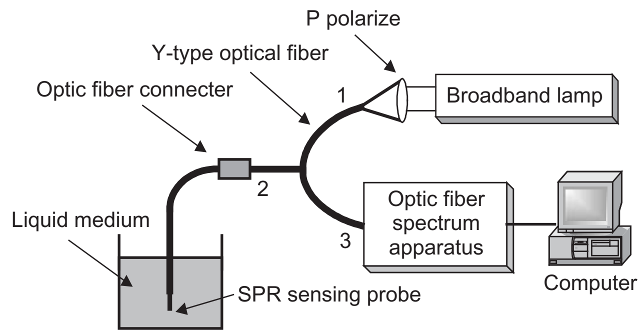

Figure 2. *Sketch of optical fiber SPR sensor system.*

waves and propagate along the surface of the metal; the energy carried by photons of light is transferred to packets of electron, the incidence light is almost completely absorbed.

One shortcoming of SPR setup based on prism is the single spot measure mode that may cause blind spots. In contrast, the different light signals transmitting in the fiber can excite SPR effect at the whole surface of the sensing fiber. This kind of excitation construction of SPR effect is the same as that of the prism type, but it forms a three-dimensional construction to excite SPR effect at the surface of the core of the fiber. In fact, there is a range of incidence angles that are allowed to propagate in the multimode fiber. The optical fiber, which can be used as the basic material, not only performs sensing, but also has the function of transmitting light. According to Maxwell formula and the complex dielectric of metal:

The wave vector of p light:

$$k_x = \frac{2\pi}{\lambda}\sqrt{\varepsilon_0(\lambda)}\sin\theta_{\mathrm{SPR}}. \tag{1}$$

The vector of surface plasmon wave, k_{sp} is given by the expression:

$$k_{\mathrm{sp}} = \frac{2\pi}{\lambda}\sqrt{\frac{\varepsilon_1(\lambda)\times\varepsilon_2(\lambda)}{\varepsilon_1(\lambda)+\varepsilon_2(\lambda)}}. \tag{2}$$

Equation (3) shows the relationship of the resonance angle with the dielectric constant of the fiber core, metal, and environmental medium (Ko et al., 2004):

$$\sin\theta_{\mathrm{SPR}} = \frac{\sqrt{(\varepsilon_1(\lambda)\times\varepsilon_2(\lambda))/(\varepsilon_1(\lambda)+\varepsilon_2(\lambda))}}{\sqrt{\varepsilon_0(\lambda)}} \tag{3}$$

where θ_{SPR} is the resonance angle, λ the wavelength of incidence light, and $\varepsilon_0(\lambda)$ and $\varepsilon_2(\lambda)$ are dielectric constants of the fiber core and the environmental medium, respectively. $\varepsilon_1(\lambda, t)$ is the complex dielectric of the metal. Equation (3) shows that θ_{SPR} is extremely sensitive to the change of $\varepsilon_0(\lambda)$, $\varepsilon_1(\lambda)$, and $\varepsilon_2(\lambda)$.

As the light signal with different wavelengths leads to dispersion effect in the fiber core and the dielectric constant of the medium layer changes with the shift of light wavelength, it is very complicated to make a precisely theoretical analysis. The relationship model between the light reflectivity and several other parameters was proposed by Niggermann and Katekamp (1996) using multilayer thin-film reflectance theory in the optical fiber SPR sensing setup.

$$R = [\theta_{\mathrm{i}}, d, l, \varepsilon_0(\lambda), \varepsilon_1(\lambda, t), \varepsilon_2(\lambda)]^{N(\theta_{\mathrm{i}}, l, d)}. \tag{4}$$

The number of reflections N at the silver surface inside the fiber is a function of the incident angle $\varepsilon_1(\lambda, t)$, the interaction length l, and the core diameter d with $N(\theta_{\mathrm{i}}, l, d)$. When the optical fiber SPR probe structure is fixed, the reflectivity can be expressed by:

$$R = f\,[\varepsilon_2(\lambda_{\mathrm{SPR}})]. \tag{5}$$

From Equation (5), it can be found that the resonance wavelength λ_{SPR} is sensitive to the small change of liquid refractive index and has a good corresponding relationship with the value of liquid refractive index.

Experimental Setup

This sensing system is schematically shown in Figure 2. A multimode optical fiber with a core diameter of 600 µm is employed as the excitation basis. The incident light is produced by the broadband lamp with a wavelength ranging from 400 to 1000 nm. It is coupled into the sensing optical fiber through the y-type optical fiber coupler after it is processed by the p-polarizer. The incidence light of a certain wavelength can interact with the liquid medium around the SPR probe. The reflected lights are recorded by the spectrum analyzer with a resolution of 0.25 nm as the integration result of the whole sensing probe interaction with the environmental medium. The curve of relationship of reflective light intensity and wavelength can be obtained by a special processing software implemented in the computer. The reflected spectrum from the SPR probe in the air would be used as reference to uniform the SPR spectrums obtained from different liquid environments.

RESULTS AND DISCUSSION

Measurements of Several Different Kinds of Liquids with the Same Refractive Index

In this experiment, two groups of solutions are made, whose refractive indices are 1.3365 and 1.4126, respectively. Each group consists of three solutions with different concentrations that are marked glycol, potash, and glycerine, respectively. Two SPR spectra, Figures 3 and 4, can be achieved by using the testing system, as shown in Figure 2. Figure 3 shows that the wavebottoms of the SPR spectrums, namely the resonance wavelength, of the three solutions with a refractive index of 1.3365 all appear at the point where $\lambda_{SPR} = 562.75$ nm. Likewise, the resonance wavelengths of the three corresponding solutions all appear at the point where $\lambda_{SPR} = 854.00$ nm, as shown in Figure 4. This phenomenon proves that when the structure of the SPW fiber optic probe is fixed, the resonance wavelength of the SPR spectrum is only determined by the refractive index of the liquid and is independent of the type of liquid. This theory can also be proved by analyzing Equation (3), which shows that the resonance angle θ_{SPR} is related to the dielectric constant of the core of the fiber optic, metal, and liquid. Only the monochromatic light wave with the total reflection angle θ_i, which is equal to the resonance angle θ_{SPR}, is capable of triggering the resonance of the surface plasma wave. At the point of resonance, the wavelength of the light wave, namely the resonance wavelength, is only determined by the refractive index of the liquid.

It is worth mentioning that though the two groups of solutions share the same refractive index, their minimum light reflectivities R_{min} are different. Figure 3 shows that R_{min} decreases in the order of glycol, potash, and glycerine, while in Figure 4, R_{min} decreases in the order of glycol, glycerine, and potash. This proves that the SPR spectrum is not subject to refractive index only. An analysis of Equation (4) shows that light intensity reflectivity is actually a function of multiple complex variables. Liquids with different types and concentrations can stimulate SPR effects with different degrees, hence the light reflectivities are different. And for this reason, both resonance wavelength and light intensity reflectivity should be taken into account when analyzing the SPR spectrum.

Optimization of SPR Probe and Experiment

Surface plasmons always exist at the interface between the high and low refractive index media. A basic principle of SPR effect for detection is that the refractive index of the core of the fiber must be higher than that of the liquid. If not, the wave vector of incidence light will always be smaller than that of surface plasmon. From Equations (1) and (2), it can be found that the enhancement of the refractive index of the core of the optical fiber will make the resonance condition become possible again.

To overcome the limitation that the refractive index of the fiber core is low, the structure of SPR probe is redesigned, as shown in Figure 5. The usual quartz optical fiber with a refractive index of 1.468 is used still as the sensing material to keep the toughness of the sensor. At first, the surface of the bore core of multimode fiber is coated with an SiO film (the refractive index is 1.85), which acts as the middle layer with a thickness of 20 μm. Then the films are covered with a silver film with a mean thickness of 50 nm.

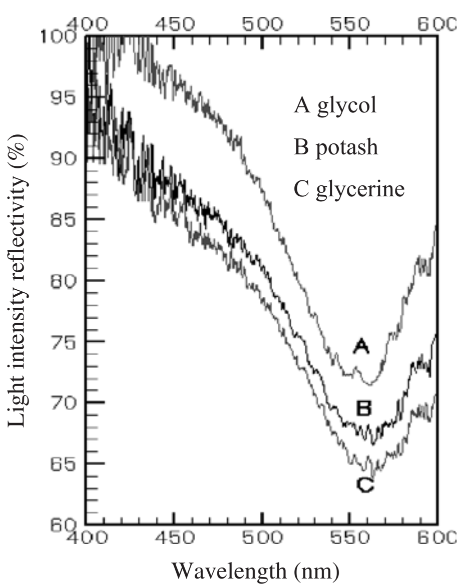

Figure 3. *SPR spectra of three different kinds of liquids with a refractive index of 1.3365.*

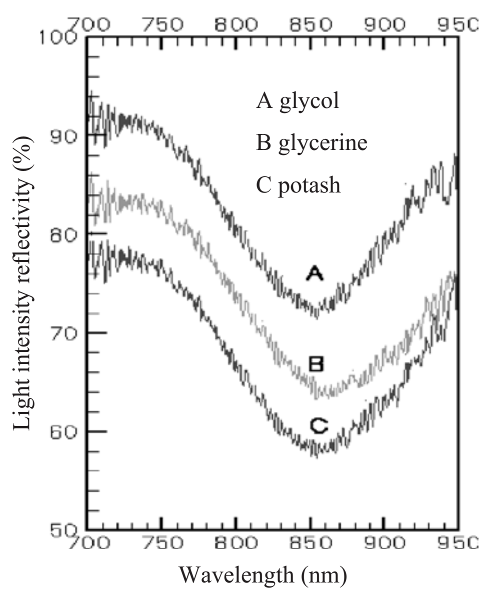

Figure 4. *SPR spectra of three different kinds of liquids with a refractive index of 1.4126.*

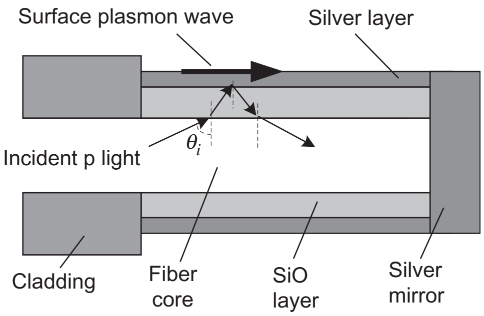

Figure 5. Structure of SPR probe coated with SiO film layer.

This method improves the value of the refractive index of the excitation medium and achieves the goal of enlarging the detection upper limit of the liquid refractive index.

In the experiments, a multimode fiber with a numerical aperture (NA) of 0.3 allows the incidence angles between 78.5 and 90°. When the incidence p light reaches the operation scope of SPR probe, it will enter into the SiO film. Surface plasmon resonance phenomenon is observed at the interface between the SiO layer and the liquid medium. An electrical density wave called the evanescent wave can leak into the liquid medium and interact with the surface plasmon of the silver film. This causes the energy carried by the photons of light to get transferred to packets of electrons called plasmon. The reflected p light can still be transmitted in the core of the fiber when it returns to the interface between the SiO layer and the silver film again due to the smaller incident angle than the critical angle for total internal reflection.

To verify the feasibility and sensitivity of this structure of probe, several liquids with a high refractive index, such as glycol, benzene, nitrobenzene, and aniline are chosen as the environmental medium; their refractive indices are 1.4344, 1.5005, 1.5520, 1.5859, respectively. To compare the results of the output, these liquids are measured by using both this probe-coated SiO film and the usual probe.

Figure 6 shows the experimental results. The spectra outputted by the usual SPR probe are the horizontal lines without any resonance wavebottom (as shown in curves E and F). This phenomenon confirms that if the liquid refractive index is higher than that of the core of the fiber, the dispersion curve of the surface plasmon wave can always not cut with that of light, that is, vector k_{sp} can always not match with k_x. On the other hand, the coated SiO film has obvious resonance wavebottom and their resonance wavelengths increase with the rise of the liquid refractive index. However, the shortcoming of this SiO film is that it can be easily scraped from the core of the optic fiber due to its thickness in the measurement process. Therefore, it is necessary to improve the plating craft and reduce the thickness of the film.

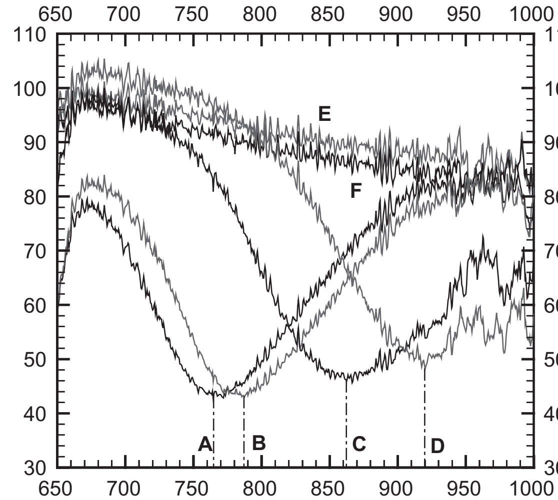

Figure 6. SPR spectra of improved probe.

CONCLUSIONS

The output characteristic of liquid refractive index measured by the optical fiber SPR sensor is discussed theoretically and experimentally. The probe structure of the optical fiber SPR, which is based on wavelength module, not only overcomes the error source caused by the unstable factor of the lamp, but also has features, such as simple structure, all light being transmitted by the fiber, and easiness to realize long distance measurement as well. When the structure of SPR probe is fixed, these liquids have the same resonance wavelength λ_{SPR} because of their same refractive indices, but their lowest light reflectivity has different values due to the diversity of their types. The knowledge about the output characteristic of the SPR spectrum can provide a new way to realize precise measurement of liquid refractive index and type identification. This improved probe shows the ability to extend the detection upper limit of refractive index. The variation of the refractive index occurring through the resin cure process will be detected by using this novel SPR probe in our next research. Curing information can be obtained by analyzing the shift of the resonance wavelength.

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REFERENCES

Alonso, R., Villuendas, F., Tornos, J. and Pelayo, J. 1993. "New In-line Optical-fibre Sensor based on Surface Plasmon Excitation," *Sensor and Actuators A*, 37:187–192.

Diez, A., Andres, M.V. and Cruz, J.L. 2001. ''In-line Optical-fibre Sensor based on Excitation of Surface Plasmon. Modes in Metal-coated Tapered Fibres,'' *Sensor and Actuators A*, 73:95–99.

Fontana, E., Pantell, R.H. and Strober, S. 1990. ''Surface Plasmon Immunoassay,'' *Appl. Opt.*, 29:4694–4704.

Homola, J. and Sinclair, S. 1999. ''Surface Plamon Resonance Sensors: Review,'' *Sensor and Actuators B*, 54:3–15.

Jorgenson, R.C., Yee, S.S. and Johnston, K.S. 1993. ''A Novel Surface Plasmon Resonance based Fibre Optic Sensor Applied to Biochemical Sensing,'' In: *Proceedings of SPIE*, Vol. 1886, pp. 35–46.

Jorgenson, R.C. and Yee, S.S. 1993. ''A Fibre Optic Chemical Sensor based on Surface Plasmon Resonance,'' *Sensor and Actuators B*, 12:213–220.

Ko, W.S., Oh, S.B. and Kim, S.H. 2004. ''Development of Fibre Type Surface Plasmon Resonance for Protein Detection,'' In: *Proceedings of SPIE*, Vol. 5272, pp. 100–108.

Miwa, S. and Arakawa, T. 1996. ''Selective Gas Detection by means of Surface Plasmon Resonance Sensors,'' *Thin Soild Films*, 6: 281–282.

Niggermann, M. and Katekamp, A. 1996. ''Intrinsic Fiber Optical Gas Sensor Based on Surface Plasmon Resonance Spectroscopy,'' In: *Proceedings of SPIE*, Vol. 2508, pp. 303–308.

Slavik, R., Homola, J. and Ctyroky, J. 2001. ''Novel Spectral Fibre Optic Sensor based on Surface Resonance,'' *Sensor and Actuators B*, 74:106–111.

Weiss, M.N., Srivastava, R. and Groger, H. 1996. ''Experimental Investigation of a Surface Plasmon-based Integrated Optic Humidity Sensor,'' *Electron. Lett.*, 32:842–843.