Theoretical Analysis of Gold-deposited Optical Fiber Sensor and Characterization of the Gold Film

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Gold-deposited optical fiber sensors with film thicknesses from 30 to 60 nm were prepared, and the responses to a wide range of a refractivity (1.33 – 1.54 refractive index (RI) units) were investigated both experimentally and theoretically. The response curve of the sensor has two minima in the refractivity range from 1.33 to 1.44 and at 1.462 RI units. The former minimum is due to surface plasmon resonance (SPR) in the thin gold film, and shifts to a lower refractivity as the film becomes thicker. The response curves of the sensors with film thicknesses of 45 and 60 nm agreed well with those calculated from SPR theoretical equations. Morphology observations of the surfaces of deposited gold films on glass by atomic force microscopy (AFM) and a variation in resistance of the films with various thicknesses show the structure of the gold films. We concluded that the thin deposited gold films have many defects, and that the core of the gold-deposited optical fiber leaks light through the defects to the sample solution with the same refractivity (1.462 RI units) as that of the core.

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Introduction

Surface plasmon resonance (SPR) is an optical phenomenon in which incident light excites a charge-density wave at the interface between a metal and a dielectric.^{1,2} Applications of SPR to optical fiber sensors have been made intensively, mainly because of the advantages of remote sensing, continuous analysis, and *in situ* monitoring with an inexpensive and disposable sensing device.^{1,2} A gold or silver film is frequently used as the metal layer of the SPR sensing element.^{1,2} Though the silver film produces sharper resonance, and consequently higher sensitivity, the surface must be protected from oxidation and corrosion. The gold film produces broader resonance than the silver film, but it is stable and inactive.

Experimental and theoretical analyses for optical fiber or waveguide sensors based on SPR were carried out, and models for the response have been proposed by many researchers.³⁻¹⁶ A typical type of the optical fiber sensor based on SPR has a thin (several tens of nanometers) deposited silver or gold film around an exposed core of a multi-mode optical fiber, and the core is used instead of a coupling prism of the SPR sensor system.³⁻⁷ These systems are constructed by modifying a traditional SPR sensor system, and scan the wavelength³ or change the angle of incident light.⁴⁻⁷ Another type of the sensor based on SPR in a thin deposited gold film on the core of a side polished single-mode optical fiber implanted in a silica block has been developed.⁸⁻¹¹ Though this type of sensor has high resolution, probably because of an optimum bend of the single-mode optical fiber, and no need to change the incident angle,

fabrication of the sensing element and the optics of the system are complicated.

Most theoretical analyses of the optical fiber sensors using SPR are based on the traditional Kretschmann configuration, and the response curves have been calculated.^{3,6-16} However, these analyses were made for the sensors in a restricted range of the refractivity (less than 1.45 RI units), and thus a theoretical investigation concerning the response of the gold-deposited optical fiber sensor in a wider range of the refractivity is necessary to understand the mechanism of the response. Morphology observations of gold films deposited on glass by a scanning tunneling microscope (STM) and atomic force microscope (AFM) have been carried out.¹⁷⁻²⁰ These studies reported that the gold film consists of small grains or islands, when it is deposited at a room temperature. Such studies provide important information on the structure of the thin gold films and also on the mechanism for the response of the gold-deposited optical fiber sensor.

We have already developed a small and simple gold-deposited optical fiber sensor system without scanning the wavelength or changing the angle of incident light. This sensor system offers numerous benefits, such as remote sensing, continuous analysis, and *in situ* monitoring.²¹⁻²⁴ We have performed analyses of various alcohol solutions²¹⁻²⁴ and some esters in ethanol²² using this sensor system. We have successfully controlled the properties of the gold-deposited optical fiber sensor by changing the film thickness.²⁴ More detailed studies in both experimental and theoretical investigations on the sensor are necessary in order to investigate the sensor properties and to understand the mechanism of the response as well as to apply it in industry.

In the present work, we investigated the response curves of the gold-deposited optical fiber sensors with various film thicknesses, both experimentally and theoretically. The surface

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morphologies of deposited gold films on glass were observed by AFM. The resistances of deposited gold films with various thicknesses on glass were measured in order to obtain information about the film structure. The properties of the sensor system are discussed along with a consideration of the morphology of the thin deposited gold film.

Principle

The gold-deposited optical fiber sensor utilizes the SPR phenomenon at the interface between a thin gold film around the core of an optical fiber and a sample solution.²¹⁻²⁴ Figure 1 shows a schematic meridional cross section of the sensing element of the gold-deposited optical fiber sensor, where θ is the incident angle of light to the interface of the core and the gold film, $I(\theta)$ is the light intensity at the incident angle θ , I is the integrated light intensity, ϵ_m is the dielectric permittivity of the gold film, and n_c and n_s are the refractivities of the core and the sample solution, respectively.

When light with an intensity distribution is introduced into the optical fiber of the sensor with a focusing lens, the input end of the optical fiber is illuminated by focused rays. The light intensity in the core depends on the incident angle θ . The incident light reflects at the inner surface of the core, and localized light, called an evanescent wave (EW), occurs. The wave number of the EW is determined by both the incident angle θ and the wavenumber of the incident light. There are many wavenumbers of the evanescent waves at the interface of the core and the gold film because of the angle range of the incident light. SPR is an optical phenomenon in which incident light excites a charge-density wave at the interface between a metal and an analyte. The EW at the interface between the core and the gold film excites the surface plasmon wave (SPW) which is propagating along the surface of the metal film and thus, a part of the incident light is absorbed. When the sample with the refractivity of n_s is introduced into the sensing element, the incident light at the incident angle θ is absorbed by the SPR excitation, and the integrated transmitted light intensity becomes $I - I(\theta)$. The actual angle range of the incident light due to the characteristics of the lens and the optical fiber used is between 70° and 90° , and the light with this angle range causes the SPR phenomenon. Thus, the refractivity of the sample surrounding the gold-deposited optical fiber can be deduced from the variation in the transmitted light intensity through the sensor.

Experimental

Sensor construction

The diameters of the core, clad, and jacket of the optical fiber (Showa Electric Wire & Cable NJ-PF200/300) used were 200, 300, and 900 μm , respectively. The clad and jacket of 10 cm long were removed by immersing the optical fiber in concentrated sulfuric acid. Gold films (Mitsuwa Chemicals 99.99%) with various thicknesses were evaporated at a rate of 0.4–0.8 nm/s around the bare cores by rotating the optical fibers every 90° in a metal bell-jar evaporator at a pressure of about 6×10^{-6} Torr (1 Torr = 133.322 Pa) and at a room temperature. The gold-deposited optical fiber and Teflon tubes for a sample inlet and outlet were fixed in a glass tube (12 cm long and 0.3 cm in diameter) with resin to form the sensor cell.

Fig. 1 Schematic representation for the principal of a response of the gold-deposited optical fiber sensor. The refractive indices of a sample solution and the core are represented by n_s and n_c , respectively. ϵ_m is the dielectric permittivity of the gold film. θ , $I(\theta)$, and I are an incident angle of the light to the inner surface of the core, the intensity of the light reflected at θ , and the integrated light intensity, respectively.

Apparatus

The sensor system was composed of a He-Ne laser (Melles Griot V05LHR151), lens (50 mm focal length), sensor cell, Si detector (ILX Lightwave VOMM-6722B), optical multimeter (YOKOGAWA 755501-1), digital multimeter (YOKOGAWA 755501-1) as an A/D converter, and computer (NEC PC-9821 Xs). Sample solutions maintained at 25°C flowed through the sensor cell at a rate of 0.098 ml/s with a micro-tube pump (EYELA MP-3N). Refractive indices of alcohol solutions were measured at 25°C with an Abbe refractometer (ATAGO DR-A1).

The surface morphologies of gold films deposited on glass substrates were observed with an AFM (Digital Instruments NanoScope III) operating in the contact mode in air. The cantilever was 200- μm long Si_3N_4 with a force constant of 0.06 N/m. The images were automatically plane-fitted to account for the sample tilt. The resistances of deposited gold films (13 mm long and 1 mm wide) with various thicknesses on glass substrates were also measured in order to investigate the film structure.

Chemicals

All alcohols (methanol, ethanol, 1-propanol, 2-propanol, 1-butanol, isobutanol, 1-pentanol, 2-pentanol, 1-hexanol, cyclohexanol, propylene glycol, 1,3-butanediol, phenethyl alcohol, and benzyl alcohol) were obtained from Wako Pure Chemical Industries and used without further purification.

Results and Discussion

Response curves of the sensors with various film thicknesses

The response curves of the gold-deposited optical fiber sensors with film thicknesses of 30, 45, and 60 nm to a wide range of refractivities from 1.327 to 1.537 RI units of methanol solutions of various alcohols are shown in Fig. 2. The transmitted light intensities for the solutions were normalized by that for the solvent at 1.327 RI units. The response of a bare core optical fiber without a gold film (0 nm) is also shown for comparison. The gold-deposited optical fiber sensors showed the broad responses due to the SPR phenomenon at the lower refractivity range from 1.33 to 1.44 RI units, whereas the bare core optical fiber had no response over this refractivity range.

Fig. 2 Response curves of the gold-deposited optical fiber sensors with the film thicknesses of 30, 45 and 60 nm to methanol solutions of various alcohols. The response curve of the bare core optical fiber without a gold film (0 nm) is also shown.

However, both types of the response curves of the gold-deposited optical fiber sensors and the bare core optical fiber showed a rapid decrease as the refractivity increased above 1.44 RI units, and had sharp minima at 1.462 ± 0.001 RI units. The light intensity gradually increased above 1.46 RI units. The transmitted light intensity of the bare core optical fiber became zero at the minimum because of a complete loss of the light from the core of the optical fiber to the solution. The value of the refractivity at the minima was very close to that of quartz (1.4572 RI units at 632.8 nm and at a room temperature). Though the cores of the sensors were covered with the thin gold films, the films were so thin (30–60 nm) that the core leaked the light to the solution due to the structure of the thin deposited gold film, as discussed below.

The minimum of the response curve due to the SPR phenomenon between 1.33 and 1.44 RI units shifted to a lower refractivity as the film became thicker. The refractivities of the minima of the response curves with the thicknesses of 30, 45, and 60 nm were 1.383, 1.374, and 1.351 RI units, respectively. The slope of the response curve also changed with the film thickness. The estimated slopes of the response curve for the sensor with the film thickness of 45 nm were -8.32 and 4.90 for the ranges of 1.33–1.36 and 1.38–1.44 RI units, respectively. These slopes have no dimension, and represent the sensitivities of the gold-deposited optical fiber sensor to the refractivities of methanol solutions of various alcohols. Thus, the sensitivity and the response range of the gold-deposited optical fiber sensor can be controlled by changing the film thickness.

The gold-deposited optical fiber sensors at an incident angle of 8° with thicknesses of 30, 50, and 70 nm have minima near 1.38, 1.35, and 1.34 RI units in the response curves, respectively.⁵ The shift of the minimum toward a lower refractivity with an increase in the film thickness has been reported in the response curves of a gold-deposited optical fiber

Fig. 3 AFM top view (top) and the section analysis (bottom) of a gold film with a thickness of 45 nm deposited on a glass substrate at a room temperature. Image size: $1 \times 1 \mu\text{m}^2$ and 20 nm gray scale. The section analysis was carried out along a line extending from the right-top corner to the left-bottom corner of the image.

sensor with the film deposited on the polished core.^{8,10,11} A shift of the minimum in the refractivity with the film thickness is a general character of these gold-deposited optical fiber sensors. These studies support our result about the shift of the minimum and the change in the properties of the gold-deposited optical fiber sensor by the film thickness.

Characterization of the deposited gold films

The surface morphologies of deposited gold films with various thicknesses on glass substrates at a room temperature were observed by AFM. The top view and the section analysis of the gold film with a thickness of 45 nm are shown in Fig. 3. The picture size is $1 \mu\text{m}$ wide and 20 nm gray scale. The section analysis was carried out along the line from the right-top corner to the left-bottom corner. The film consists of small round grains with a diameter of about 30 nm. The film is typical polycrystalline and the structure is characterized by columns with domed tops.^{17–20} An average grain size of about 30 nm has also been observed for a gold film deposited on glass at a room temperature by STM.¹⁸ The surface morphology of the film is caused by self shadowing during deposition, because the surface diffusion of adatoms is too small to allow migration into the shadow regions at this temperature.^{25–30} The roughness analysis gives the root-mean-square roughness (R_{ms}) of about 1 nm, and the value becomes larger with an increase in the film thickness, as reported previously.^{18,20} The section analysis indicates that there are spaces between grains; the average depth of the grain boundaries is about 5 nm. The maximum height between the highest and lowest point (R_{max}) in the image is about 10 nm. These depths are smaller than the film thickness. However, it is known in AFM that the finite size of a tip may distort the resultant profiles of finely structured surfaces.^{25–30} The distortion due to the finite tip size (20 to 40 nm)³¹ makes the spaces between grain boundaries or voids of the film smaller than they actually are.^{25–28} We conclude that the image of the gold film does not show smaller features less than the tip size. The depth of grain boundaries in the section analysis is shallower than a real depth. Much smaller features, such as holes or cracks, do not appear in the AFM image. Thus, some

Fig. 4 Resistances of deposited gold films with various thicknesses on glass substrates. The length and width of the gold films were 13 and 1 mm, respectively.

grains may be completely separated and the boundaries actually arrive at the substrate.

The resistances of deposited gold films with various thicknesses on glass substrates are shown in Fig. 4. Schematic representations of the gold films are also shown in the figure. The length and width of the gold film were 13 and 1 mm, respectively. The resistances of films thinner than about 15 nm rapidly increased as the thickness decreased. The slope of the straight line was -4.4 . This is because the film has an isolated island structure, and electrons are transferred between the islands by a tunneling phenomenon or a thermionic emission. On the other hand, the resistance decreased inversely proportional to the film thickness above 15 nm, as expected by a metal conduction mechanism. The slope of the straight line was -1.0 . The resistance variation of the gold films on glass indicates the electrical continuity of the film with the thickness above 15 nm. He and Shi¹⁹ observed gold films deposited on glass at a room temperature, and found an island morphology and a high density of grain boundaries by AFM. They have also reported that the gold film becomes conducting above 10 nm. It is clear that thin gold films deposited on glass have many defects, such as channels and voids. Thus, we conclude that the deposited gold films with thicknesses of 30–60 nm on the quartz core of the optical fiber are electrically continuous, but have many defects. The light leaks from the core through the defects of the deposited gold film to the solution with the same refractivity as that of the core of the optical fiber.

Theoretical analysis of the response curves

The principal of the metal-deposited optical fiber sensors is based on the Kretschmann configuration in SPR and models of the response have been proposed.^{3,6-11,14,15} Jorgenson and Yee³ replaced the prism by a multi-mode optical fiber and applied the traditional SPR theory to the silver-deposited optical fiber sensor. Trouillet *et al.*⁶ and Lin *et al.*⁷ developed a three-dimensional model of a multi-mode optical fiber covered with a silver or gold film, and concluded that the light energy transmitted out of the fiber is not affected by the polarization of the incident light. Liu *et al.*¹⁴ calculated the reflectance curves of a multi-mode optical fiber covered with a silver film using a multiple-reflection theory, and also considering an angle range of incident light. Xu *et al.*¹⁵ calculated the optical power transmittance of a gold-deposited multi-mode optical fiber

sensors as a function of the wavelength. Alonso *et al.*⁸ and Homola *et al.*⁹⁻¹¹ carried out theoretical analyses of gold-deposited sensors of a side polished single-mode optical fiber based on an equivalent planar waveguide approach. Čtyroký *et al.*^{12,13} also calculated the relative power transfer of a waveguide SPR sensor with various thicknesses of gold films. Kurihara and Suzuki¹⁶ recently provided a general and practical theory of optical-absorption-based SPR using a three-layer Fresnel equation. This theory may be applicable to future development in metal-deposited optical fiber sensors.

We carried out the numerical calculations of the response curves of our gold-deposited optical fiber sensors based on the theoretical analysis of the optical power transmittance of the optical fiber SPR sensor made by Xu *et al.*¹⁵ They assumed that the theoretical models of SPR for bulk optics are satisfied by meridional rays in optical fibers as shown in Fig. 1. In our calculation, we also considered the meridional ray, and assumed single reflection without a range of incident angles in the multi-mode optical fiber for simplicity.

The propagation constant of the EW is expressed as¹⁵

$$k_{ev} = \frac{2\pi}{\lambda} n_c \sin \theta, \quad (1)$$

where λ is the wavelength, n_c is the RI of the core, and θ is the incident angle. The propagation constants of the SPW are expressed by the following equations:

$$k_0 = \frac{2\pi}{\lambda} \left(\frac{\epsilon_s \epsilon_m}{\epsilon_s + \epsilon_m} \right)^{1/2}, \quad (2)$$

$$k_r = k_0 \left(\frac{2}{\epsilon_s + \epsilon_m} \right) \left(\frac{\epsilon_s \epsilon_m}{\epsilon_s + \epsilon_m} \right)^{3/2} \exp \left\{ i \frac{4\pi d}{\lambda} \frac{\epsilon_m}{(\epsilon_s + \epsilon_m)^{1/2}} \right\}, \quad (3)$$

where k_0 is the dispersion relation of SPR of a metal-vacuum interface in a one-sharp-boundary model, k_r is the perturbation to k_0 due to the finite physical volume of the metal, ϵ_s is the dielectric permittivity of a sample approximated by $\epsilon_s = n_s^2$, ϵ_m is the dielectric permittivity of gold, and d is the film thickness.

When Eqs. (1), (2), and (3) satisfy the following equation, the SPR phenomenon occurs and a part of incident light is absorbed:

$$k_{ev} = k_0 + k_r. \quad (4)$$

Equations from (1) to (4) can be transformed to

$$\sin \theta = \frac{1}{n_c} \left(\frac{\epsilon_s \epsilon_m}{\epsilon_s + \epsilon_m} \right)^{1/2} \times \left[1 + \left(\frac{2}{\epsilon_s + \epsilon_m} \right) \left(\frac{\epsilon_s \epsilon_m}{\epsilon_s + \epsilon_m} \right)^{3/2} \exp \left\{ i \frac{4\pi d}{\lambda} \frac{\epsilon_m}{(\epsilon_s + \epsilon_m)^{1/2}} \right\} \right]. \quad (5)$$

The resonance angle θ is obtained from Eq. (5) and is a function of λ , d , n_s , and ϵ_m .

The intensity distribution for an incident angle θ of the collimated laser light focused on the end of the optical fiber is expressed as¹⁵

$$I(\theta)_{\infty} = \frac{n_c^2 \sin \theta \cos \theta}{(1 - n_c^2 \cos^2 \theta)^2}. \quad (6)$$

The reflectivity at the interface between the core of the optical fiber and the sample is given by the Fresnel equation of p-polarized light, as follows:

$$R_p = \left| \frac{\frac{\cos \theta}{n_s} - \frac{\cos \theta'}{n_c}}{\frac{\cos \theta}{n_s} + \frac{\cos \theta'}{n_c}} \right|^2, \quad (7)$$

where θ and θ' are the incident angle to the inner surface and the refraction angle to the outer surface of the core, respectively.

The numerical calculations of the response curves due to SPR were performed for various values of d . The parameters used for the calculations were: $n_c = 1.4572$, $\epsilon_m = -11.836107$,¹³ and $\lambda = 632.8$ nm. The resonance angle θ was given first by Eq. (5), and then the transmitted light intensity was calculated by subtracting the light intensity absorbed due to SPR between the angle range of $\theta \pm \Delta\theta$ from the total intensity of Eq. (6). Though the full width at half maximum (FWHM) of a peak in the SPR reflection spectrum of a gold film is generally wider than that of a silver film, $\Delta\theta = 1^\circ$ was used because the FWHM of a peak in a theoretical SPR reflection spectrum of a silver film is about 2° .^{3,6} The calculated transmitted intensity was normalized by the total intensity.

The incident angle θ of Eq. (7) was calculated using the focusing length of the lens and the diameter of the laser beam. The actual incident angle θ is considered to have a distribution between 70° and 90° . However, the calculations of the reflectivity at the interface between the core and the sample, given by Eq. (7), were carried out for the incident angle $\theta = 88^\circ$ for simplicity, and θ' was calculated by the Snell law from the refractive indices of the core and a sample. The value of R_p was assumed to be 1 in the range of the total reflection below 1.4572 RI units. The theoretical curve was finally obtained by the product of the transmitted intensity calculated from the SPR equations and the reflectivity calculated from the Fresnel equation. The numerical calculations were made for every 2×10^{-5} RI units.

The theoretical response curves of the gold-deposited optical fiber sensors with the film thicknesses of 30, 45, and 60 nm are shown in Fig. 5. These curves for the sensors with the film thicknesses of 30, 45, and 60 nm have minima due to the SPR phenomenon at 1.448, 1.369, and 1.350, respectively. The theoretical curves are sharper than the experimental curves shown in Fig. 2. This is mainly caused by the assumption of single reflection without a range of incident angles in spite of multi-reflection and the range of incident angles in the multi-mode optical fiber. The intensity distribution of the laser used in the theoretical calculation may be narrower than the actual distribution. Though the theoretical curve due to SPR became broad with an increase in the value of $\Delta\theta$, the position of the minimum in the curve calculated for $\Delta\theta = 2^\circ$ did not change of the order of 10^{-3} RI units.

The theoretical response curves show a shift of the minimum to a lower refractivity as the film becomes thicker. The positions of the minima of the theoretical curves of 45 and 60 nm agree well with those obtained in the experiment (1.374 and 1.351 RI units, respectively). In the case of 30 nm, however, the position of the minimum in the theoretical curve disagrees with that (1.383 RI units) in the experimental curve. This is probably due to the difference between the actual and measured thickness of the deposited gold film with a columnar structure, as shown in Fig. 3. Since the thickness was measured by the mass with the crystal frequency monitor, the actual thickness of

Fig. 5 Response curves of the gold-deposited optical fiber sensors with the film thicknesses of 30, 45, and 60 nm calculated from the SPR theoretical equations and the Fresnel equation. The response curve of the bare core optical fiber (0 nm) calculated from the Fresnel equation is also shown for comparison.

the film with the columnar structure is slightly thicker, and thus, the difference in the film thickness may have an influence on the SPR in the case of the thickness of 30 nm. The dielectric constant of a very thin gold film may be different from that of the thicker gold films used in the present theoretical calculations, and thus, may cause a difference in the SPR phenomenon. Therefore, it is important to obtain information about morphology of very thin gold films for a theoretical analysis and understanding the response of the gold-deposited optical fiber sensor.

The sharp minimum at 1.457 RI units in the theoretical curve exists at a slightly lower refractivity than that (1.462 ± 0.001 RI units) of the experimental curve. It seems that the actual RI of the core is higher than that of quartz. The calculation was carried out again for $n_c = 1.462$ RI units at about the film thickness of 45 nm, and the value of the minimum in the calculated response curve due to SPR was 1.373. The good agreement suggests the actual value of the RI of the core of the optical fiber.

The theoretical analyses of the response curves of the gold-deposited optical fiber sensors show the mechanism for the response. The former minimum below 1.45 RI units is due to the maximum excitation of SPR in the deposited gold film, and the position depends on the thickness. The latter minimum at 1.462 RI units is caused by the leakage of light from the core to a sample solution through defects in the thin deposited gold film.

Conclusions

The response curves of the gold-deposited optical fiber sensors with the thicknesses of 30, 45, and 60 nm have the broad and sharp minima at 1.351–1.383 and 1.462 ± 0.001 RI units,

respectively. The morphology observations of the deposited gold films by AFM and the variation in the resistance of films with various thicknesses showed that the deposited gold film is electrically continuous, but has many defects. The former minimum at 1.351 – 1.383 RI units is caused by the maximum excitation of SPR at the interface between the gold film and the sample solution. The latter minimum at 1.462 RI units is due to the loss of light from the core to the solution with the same refractivity through defects in the deposited gold film. The response curves calculated from the SPR theory and the Fresnel equation agreed well with the experimental curves, and support the above conclusion.

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