Performance Enhancement of the Surface Plasmon Resonance Sensor Through the Annealing Process

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ABSTRACT
An improved Drude-Lorentz model was proposed to describe the effect of annealing on the surface Plasmon resonance (SPR) sensor. In the new model, the grain structure is taken into account to describe theoretically the dispersion of gold films. Then the improved model is used to analyze the performance of SPR sensors before and after annealing. The results indicate that the grain structure of the gold film affects the resonance properties significantly. In our research, a considerable improvement in SPR fiber sensor performance is observed after annealing. The grain size of the gold film rises from 10.8 nm to 34.3 nm, and the absorption spectrum of the SPR fiber sensor becomes deeper and sharper, with the figure of merit increasing from 18.5 to 22.8 after annealing. Comparing with other sensor performance enhancement methods, the annealing process does not change the sensor structure or add new modified layer. Therefore, most SPR sensors can be further annealed to improve the quality of the sensor after fabrication. This property makes the application of annealing very promising in optical sensing region.

INDEX TERMS
Fiber optic sensor, annealing, Drude relaxation rate, grain boundaries effect, surface plasmon resonance.

I. INTRODUCTION
Surface Plasmon resonance (SPR) effect is a resonant oscillation of the conduction electrons at the interface between the negative and positive permittivity materials stimulated by the incident light. The optical fiber SPR sensor is a novel bio-chemical sensor which combines the optical fiber technology and SPR effects. Generally, the materials with low Drude relaxation rate, high plasma frequency, and high-frequency inter-band transitions have been widely used in the SPR sensor. Therefore, gold and silver are better choices for the SPR sensor. Compared with silver, gold has better chemical stability and biological affinity, so it is widely used in biological and chemical sensing region [1], [2]. However, gold has a higher Drude relaxation rate than silver, which causes a drop in the SPR sensor quality.

To improve the detection accuracy of the SPR sensor, many performance-enhancement schemes have been proposed, for example, changing the sensor structure to increase the sensor sensitivity [3], enhancing the SPR sensing substrate by a multilayer structure based on modified materials [4], or combining the localized SPR excitation based on noble-metal nanomaterials [5], [6]. In this paper, we try to improve the performance of the gold based SPR sensor by the annealing process. This method does not change the sensor structure or add new modified layer. Previous works have shown that the annealing process can cause significant internal grain growth and surface roughness increase [7]. The electrical conductivity of metal films improves after annealing [8]. The performance of the gold nanoantennas increases due to the growth of the grain size [7]. Some researchers have studied the effects of annealing on the surface morphology of gold film. After annealing, the surface roughness of the gold film increased and the SPR absorption peak is blue-shifted [9], [10].
In addition, annealing also increases the biocompatibility of the optical sensor [11]. But qualitatively discussions between grain growth and SPR absorption spectrum have not been performed. This effect becomes more important because the annealing process can improve the sensor performance with other sensitivity enhancement methods synergistically. In the work reported here, the effect of annealing on SPR sensors is described accurately through a series of experimental measurements and theoretical studies.

Permittivity of the metal film is a key factor need to be monitored during annealing process. The Drude-Lorentz model (DL model) usually used to describe the permittivity of bulk metal [12]. However, recently researches based on the simulations and experimental results have shown that the optical property of the metal nano-films (in the range of 16-70nm) is greatly different with bulk materials [13]–[15]. The DL model does not consider some important scattering mechanisms of thin films, such as electron-surface scattering and electron-grain boundary scattering. In order to consider the effect of grain growth of the sensor during annealing process, an improved DL model was introduced.

In this paper, the effect of grain boundaries on the SPR absorption spectrum was researched, to our knowledge, for the first time. A series of fiber SPR sensors were prepared by using a magnetron sputtering system. The annealing process is used to change the grain structure of the gold film. After annealing, the geometric dimensions and the grain growth of SPR sensors were characterized by atomic force microscopy (AFM) and X-Ray Diffraction (XRD). The transmission spectrum of SPR sensors before and after annealing was measured by an SPR sensing system. An improved DL model was introduced to verify the results of the annealing experiments. The experimental results and the simulation results have been agreed well with each other. All these results indicate that the annealing process can improve the performance of SPR sensors.

II. THEORY

The classical coupling condition of surface Plasmon can be written as [3]:

\[ N_{sp} = \sqrt{\varepsilon_r n_t^2 - \varepsilon_r n_s^2} \]  

(1)

where \( N_{sp} \) is the effective index of the surface plasmon, \( n_t \) is the refractive index (RI) of the sensing layer. In Eq. (1), the complex dielectric function \( \varepsilon_r \) of the gold film can be expressed by DL model [12], [16]:

\[ \varepsilon_r(\omega) = 1 - \frac{f_0 \omega_p^2}{\omega^2 + i \Gamma_0 \omega} + \sum_{j=1}^{k} \frac{f_j \omega_p^2}{(\omega_j^2 - \omega^2) + i \Gamma_j \omega} \]  

(2)

The DL model has two terms: the Drude term for free-electron resonance and the sum of the Lorentz terms which represents the inter-band transitions. In Eq. (2), \( \omega \) is the angular frequency of the incident light, \( f_0 \) denotes the strength of Drude term, \( \omega_p \) represent the plasma frequency. The total damping term \( \Gamma_0 \) is the sum of the collision rate from electrons-electrons \( \omega_{ce} \) and electrons-phonons \( \omega_{cp} \) in the bulk metals, and \( k \) denotes the number of the oscillators at the frequency of \( \omega_j \), the strength of \( f_j \), and the life time of \( 1/\Gamma_j \).

The DL model clearly describes the dielectric function of bulk metals as the function of light frequencies. However, when the film thickness closes to the electron mean-free-path (for gold bulk material, the electron mean free path is 38 nm at room temperature [17]), the electron-grain boundary scattering has a significant effect on the total damping term \( \Gamma_0 \). In order to analyze the effect of the grain boundaries, we introduced a quantitative model based on the linearized Boltzmann equation [18]. In this quantitative model, the grain boundaries are represented by \( N \) parallel planes, with an average separation \( d \). If \( d \) is identified with the average grain diameter \( D \), the model gives the total damping term \( \Gamma_g \) in the metal film with grains:

\[ \Gamma_g = \frac{\Gamma_0}{1 - 1.5 \beta + 3 \beta^2 - 3 \beta^3 \ln(1 + \beta^{-1})} \]  

where

\[ \beta = \frac{v_F R}{\Gamma_0 D(1 - R)} \]  

(3)

where \( \beta \) is an dimensionless and intermediate variable, \( v_F \) is the Fermi velocity for gold. \( R \) represents the electron reflection coefficient at the grain boundaries, and \( D \) denotes the mean grain diameter of the film material. The model described in Eq. (3) has considered the grain boundary scattering mechanism in Drude relaxation rate. In Eq. (2), by replacing bulk metals total damping term \( \Gamma_0 \) with \( \Gamma_g \), we can get an improved DL model (called DL-MS model), which considers the effect of the grain boundaries on the thin gold films.

The permittivity of gold film can be calculated by DL-MS model, which considered the effect of grain boundaries, using the parameters provided in table 1 [18], [19]. The imaginary parts of the gold film permittivity for different \( D_s \) and different \( R_s \) are shown in Fig. 1.

It can be seen from Fig. 1(a) that, with \( D \) increasing, the imaginary part of the permittivity decreases observably. Besides, Fig. 1(b) shows that, with \( R \) increasing, the imaginary part of the dielectric function increases observably. That is because the increase of grain size and the decrease of \( R \) will reduce the gold film Drude relaxation rate significantly, leading to the decrease in the permittivity imaginary part of the gold film. It should be noted that, since the real part of the permittivity was changed very slight, so there is no separate figure about it.

After the effect of grain boundaries on the gold film has been discussed, we established an SPR sensor model to calculate the transmission spectrum of the SPR sensor. In an optical fiber SPR sensing setup, the relationship between the effective transmitted power and several other parameters can...
be written as [20]:

\[ P_{\text{trans}} = \frac{\int_{\alpha_{c}}^{\pi} R_{\text{re}}(\alpha, l, d_{\text{fiber}}) n_{s}^{2} \sin \alpha \cos \alpha \frac{n_{s}^{2} \sin \alpha \cos \alpha}{(1-n_{s}^{2} \cos^{2} \alpha)^{2}} d\alpha}{\int_{\alpha_{c}}^{\pi} n_{s}^{2} \sin \alpha \cos \alpha \frac{n_{s}^{2} \sin \alpha \cos \alpha}{(1-n_{s}^{2} \cos^{2} \alpha)^{2}} d\alpha} \]  

where \( R_{\text{re}} \) is the function of the TM- polarized light incident angle \( \alpha \), the interaction length \( l \), the fiber core diameter \( d_{\text{fiber}} \), the fiber core dielectric function \( \varepsilon_{0} \), sensing layer RI \( n_{s} \), and gold film dielectric function \( \varepsilon_{r} \). \( R_{\text{re}} \) is the light reflectivity after once reflection on the fiber-gold interface and can be calculated using multilayer thin film reflectance theory [20], [21]. \( N(\alpha, l, d_{\text{fiber}}) \) is the number of reflections at the gold surface inside the fiber, which is the function of \( \alpha \), \( l \) and \( d_{\text{fiber}} \). \( \alpha_{c} = \arcsin(n_{s}/\sqrt{\varepsilon_{0}}) \) is the critical angle of the fiber. We choose an SPR sensor with the optical fiber diameter \( d_{\text{fiber}} = 0.6 \text{mm} \), the length of the sensor region \( l = 10 \text{mm} \) and the thickness of the Gold film \( d_{\text{Au}} = 50 \text{nm} \) to simulate the effect of the annealing process. The permittivity of Gold film is calculated by the DL-MS model. The absorption spectrum is calculated by the wavelength interrogation method [22]. A series of SPR curves with different \( R_{s} \) and \( D_{s} \) are shown in Fig. 2. It can be seen that, with the increasing of \( D \) and the decreasing of \( R \), the depth of the SPR curve increases obviously, while the full width at half maximum (FWHM) decreases a lot. This is because the grain growth of the gold film leads to a decrease in the Drude relaxation rate, thus the photoelectric characteristics of the gold film are improved significantly. The quality of the final SPR sensor is also improved. One thing to note is that the grain growth of the gold film does not increase the sensitivity of the sensor. This is because the increase of \( \varepsilon_{r} \) will lead to the imaginary part of \( \varepsilon_{r} \) increase, and the real part of \( \varepsilon_{r} \) will remain unchanged. This means that the grain growth of the gold film does not affect the dispersion relation of the SPR system. Therefore, the deposition position of the SPR curve is not affected by the grain growth.

We cannot increase the sensor performance indefinitely through the annealing process. When \( D \) is small and \( R \) is high, the free electrons in the gold film are mainly affected by the grain boundary effect, and the grain boundary scattering is the main mechanism in various scattering mechanisms. The growth of the grain structure will increase the Drude relaxation rate and improve the sensor performance significantly. However, when the grain size is equal to or larger than the...
mean free path of electrons, the grain boundary scattering will be smaller than other scattering mechanisms. The effect of grain growth on the SPR sensor performance can be negligible. To further understand the effect of grain boundaries on SPR sensors, a scan of $D$ and $R$ for the absorption peak depth and FWHM of the SPR sensor are shown in Fig. 3. It can be seen from Fig. 3(a) that the most obvious drop of SPR absorption peak occurs at $D$ ranges from 10nm to 25nm, with $R$ range from 30% to 50%. It can be seen from Fig. 3(b) that the FWHM of the most obvious drop for the SPR curve is at $D$ ranges from 10nm to 30nm, with $R$ from 20% to 50%. The simulation results in Fig. 3 confirm the existence of “annealing sensitive range” for fiber SPR sensors. Annealing can improve the quality of the sensor effectively in the annealing sensitive range, out of this range the effect of annealing is negligible. These results are also consistent with the experiment results.

III. SENSOR MANUFACTURE AND SYSTEM

The manufacture of the fiber SPR sensor mainly used the following materials: polymer cladding silica (PCS) optical fiber with numerical aperture of 0.37 and core diameter of 0.6 mm (purchased from Scitlion), gold target (99.999% pure, purchased from ZhongNuo Advanced Material). And the reagents as acetone and ethanol (analytically pure grade) were purchased from local suppliers.

To fabricate a SPR probe, we selected 10 mm at the center of the PCS fiber to remove the cladding and coating apart, and then used acetone and ethanol to clean the unclad portion in the ultrasonic cleaner. After preprocessing, the gold film with thickness of 50 nm has been coated on the circumferential surface of the unclad portion by Magnetron Sputtering System. The fiber will rotate continuously during sputtering to ensure the gold film thickness was uniform around the circumference of the fiber. The vacuum of the sputtering space before coating is $4 \times 10^{-5}$Pa and the deposition rate is 0.26 nm/s of the gold film. The prepaid SPR sensor is shown in Fig. 4(a). In order to obtain SPR sensors with different grain sizes, the SPR sensors have been placed in muffle furnace annealing for 15 minutes at 100°C, 200°C, 300°C and 400°C respectively. We chose such conditions because the excessive long annealing time and the excessive high annealing temperature can lead to unexpected changes for the gold film, such as cracking and melting on the gold film surface [7], [15]. After annealing process, the transmission spectrums of the SPR sensor are measured by the SPR sensing system. The sensing system shown in Fig. 4(b) has three parts. Circular polarized light from the broadband light source (HL-2000, Ocean Optics) is injected into the multimode fiber and passed through the SPR probe, which is placed in pure water. Then the transmitted light is measured by...
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FIGURE 6. AFM 3D images of the gold film before annealing (a), after annealing at 200°C (b), after annealing at 400°C (c). (The image size is 2µm x 2µm).

The spectrograph (model HR 4000, Ocean Optics) with the detecting range of 200-1100 nm and the resolution of 20 pm.

IV. ANNEALING EXPERIMENT AND ANALYSIS

Annealing with gold film can cause significant internal grain growth, accompanied with surface roughness increasing and geometric dimensions changing. This process can be described by the grain boundary migration [23], [24]. After the SPR sensor was annealed at different temperatures, we used XRD (D/MAX 2500, Rigaku Corporation) to analyze the internal metal grain growth of gold film, use AFM (afm5500, Agilent) to measure the surface appearance of gold film.

For XRD measuring, the grain size $D$ is determined from the (111) reflections in the $\theta/2\theta$ mode by using the standard Debye-Scherrer formulation [25]:

$$D_{(111)} = \frac{\kappa \lambda}{\cos \theta \sqrt{B_{obs}^2 - B_{std}^2}}$$  \hspace{1cm} (6)

In Eq. (6), $D_{(111)}$ is the average thickness of the grain perpendicular to the grain plane. We assumed that $D_{(111)} \approx D$, $\kappa$ is the Scherrer constant, $\lambda$ denotes the X-ray wavelength, $\theta$ is the diffraction angle, $B_{obs}$ represents the FWHM of the measured sample and $B_{std}$ equals 0.19° as measured for Gold (111) single crystal standard [26]. In our experiments, $\kappa$ is set to 0.9, which is typical for spherical grains, $\lambda$ is 0.154 nm for copper anode.

Fig. 5 shows the XRD reflection intensities of the gold film before and after annealing. It can be seen that, with the annealing temperature increase, FWHM of the X-ray reflection peak becomes sharper. The slight angle shift in Fig. 5 seems to be caused by the stress changes of the gold film during the annealing process. According to Fig. 5 and Eq. (6), the grain size of gold film can be calculated. The grain size of the gold film before annealing is 10.8 nm. After annealing at 200°C, the grain size grows to 29.8 nm. While at 400°C, it grows to 34.3 nm. This result illustrates that the proper annealing time and the annealing temperature causes significant grain growth of the gold film.

We used AFM to analyze the surface morphology of the gold film. In Fig. 6(a), the Root-Mean-Square (RMS) roughness of the gold film before annealing is 0.46 nm, the small bumps signify the formation of small grains. In Fig. 6(b), RMS roughness rises to 1.29 nm after annealing at 200°C. In Fig. 6(c), RMS roughness rise to 2.06 nm after annealing at 400°C. That is because the annealing can create a larger grain, moreover, with the annealing temperature increases, some grain boundaries meet on the free surface, which leads to bigger average roughness.

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The SPR absorption spectrums are measured by the SPR sensing system. Fig. 7(a) shows the SPR curves before and after annealing at 200°C. The depth of the absorption peak has declined by 3.25%, while the FWHM of SPR curve declined by 13.32 nm. Fig. 7(b) shows the SPR curves before and after annealing at 400°C, the depth of the absorption peak has declined by 8.53%, while the FWHM of SPR curve declined by 39.6 nm.
peak has been declined by 3.63%, while the FWHM has been declined by 18.14 nm. The experimental results show that the SPR curve becomes deeper and sharper after annealing. The reason seems to be the growth of grain size. With the grain size grows, the Drude relaxation rate of the gold film decreases, which leads to a decrease in the imaginary part of the dielectric function. For SPR sensors, the reduction in the imaginary part of the dielectric function of the gold film leads to a deeper and narrower SPR absorption peak ultimately. In addition, the blue shifts of the SPR curves after annealing at 200°C and 400°C are caused by the surface roughness increase of the gold film. This effect has been simulated in Ref. [7], and the results shows that small amount of roughness change of the gold film may result in the SPR curve blue shifted.

Figure of merit (FOM) is a physical quantity that evaluates the quality of a sensor, which is defined as $FOM = S / \text{FWHM}$. Where $S$ is the RI sensitivity of the sensor. Taking into account that it is easier to measure the exact location of a narrow resonance than a broad one, larger value of FOM means a better performance of the sensor. For SPR sensor, FOM is the ratio between the wavelength shift sensitivity and the FWHM of the resonance. When the SPR sensor annealing at 400°C, FOM increasing from 18.25 to 22.8. FOM quantifies the effect of annealing on the SPR sensor. In the end, the main parameters of the two samples are shown in Table 2.

In order to get the most suitable annealing temperature, we measured the SPR sensor annealing at 100°C, 200°C, 300°C and 400°C for more refined analyses. From Fig. 8 we can know that, as the annealing temperature increases, the SPR curve becomes deeper and sharper, but when the annealing temperature rising to 300°C, this change is no longer significant. Because after the grain size rising to the electrons mean free path, the electron-grain boundaries scattering becomes quite weak, and consequently the SPR curve is slightly affected by annealing process.

### Table 2. The experimental results of the optical fiber SPR sensors before and after annealing.

| Annealing temperature | Before | After | Before | After |
|-----------------------|--------|-------|--------|-------|
| 200°C                 |        |       |        |       |
| Absorption wavelength (nm) | 660.8 | 609.9 | 659.3  | 600.2 |
| Absorption peak depth (%) | 69.54 | 66.29 | 68.98  | 65.35 |
| RMS Roughness (nm) | 0.46 | 1.29 | 0.46 | 2.06 |
| Grain size (nm) | 10.8 | 29.8 | 10.8 | 34.3 |
| FWHM (nm) | 100.58 | 87.26 | 99.26 | 81.12 |
| Sensitivity (nm/RIU) | 1816.3 | 1822.4 | 1836.6 | 1846.8 |
| FOM (A.U.) | 18.1 | 20.9 | 18.5 | 22.8 |

### V. CONCLUSION

In summary, we proposed a DL-MS model to describe the effect of annealing process on SPR sensors. After annealing at 400°C, the transmission spectrum of the SPR sensor becomes deeper and sharper, the FOM of SPR curve increases from 18.5 to 22.8. The DL-MS model was suitable to explain the experimental results. All the conclusions prove that the annealing process can significantly improve the quality of SPR sensors without changing the structure of sensors. Therefore, theoretically, most SPR sensors can be further annealed to improve their quality of the sensor after the fabrication. This property makes the application of annealing very promising in optical sensing region.

However, we should recognize that when the grain size of gold film is approach to or larger than the electrons mean free path, the effect of electron-grain boundaries on the SPR curve can be negligible. That means there is a limitation to improve the performance of SPR sensor.

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