In-Situ Thickness Measurement System for Porous Alumina Film Based on AFM and Spectrometer

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Abstract. An atomic force microscope (AFM) and spectrometer combined system for in-situ thickness measurement of nano-porous alumina (PA) films is introduced. The AFM is applied to obtain the porosity of PA film, and then we calculate the effective refractive index by Maxwell-Garnett effective dielectric constant theory. The optical thickness of PA film is determined by reflective interference spectrometry. The PA film thickness can also be measured via scanning the crosssection with optical microscope and scanning electron microscope (SEM). The sample will be damaged, however, when using this method. The measurement by optical microscope is convenient, but it’s hard to attain high resolution. When using SEM, the sample surface must be gilt and needs a vacuum environment. The system we developed can avoid the disadvantages of the method. It realizes non-destructive and high-resolution in-situ measurement.

1. Introduction
When an aluminum chip is electrochemical anodic oxidated, a layer of porous alumina film will grow on its surface [1]. The porous alumina film has unique structure, which is well-arranged hexagonal cell arrays, and there is a nano-pore in the center of every cell. These pores with uniform size are parallel with each other and vertical with the substrate. The pore diameter ranges between several nanometers and hundreds nanometers according to the oxidation condition, and the thickness of PA film between dozens nanometers and dozens micrometers. Recently, PA films have been used to prepare nanostructures for a wide range of applications [2-3].

The thickness of the nano-PA film is an important factor. Determination of the film thickness via scanning the crosssection with optical microscope or scanning electron microscope (SEM) is conventional methods. The sample must be damaged when using this method. The measurement by optical microscope is convenient, but it is hard to attain high resolution. When using SEM, the sample surface must be gilt and needs a vacuum environment. The measurement system based on AFM and reflective spectrometer we developed avoids the disadvantages of these two methods. It realizes non-destructive and in-situ thickness measurement of PA film, and has high-resolution.

2. Method and equipment
The schematic diagram of the system equipment for PA film measurement is shown in Figure 1. The system consists of AFM, fiber spectrometer, computer and some holding fixtures. The optical thickness could be determined according to the reflective spectrum. The porosity is obtained from the
AFM image, and then we calculate effective refractive index by Maxwell-Garnett effective dielectric constant theory [4]. The actual thickness of PA film would be determined eventually.

2.1. Determination of the optical thickness by reflective spectrum

The spectrometer used in this system is Ocean Optics Inc.’ HR2000 series high-resolution fiber optic spectrometer, which provides optical resolution as good as 0.065 nm (FWHM). The HR2000 is responsive from 200-1100 nm. The HR2000 Spectrometer connects to a notebook or desktop PC via USB port or serial port. When connected to the USB port of a PC, the HR2000 draws power from the host PC, eliminating the need for an external power supply.

The principle of the optical thickness measurement based on reflective interference spectrum is shown in Figure 2. When a white light beam illuminates the surface of PA film, the reflective light beams from the front and the rear interfaces are coherent, and appear regular and fine reflective spectrum. For the light whose wavelength is \( \lambda_i \), the optical path difference of reflective light beams from the front and the rear interfaces is \( 2nd\cos \theta \). Given by Bragg Equation

\[
2nd\cos \theta = m_i\lambda_i
\]

where \( n \) is the effective refractive index, \( d \) the thickness of PA film, \( m_i \) the order number with integer values for maxima and half-integer values for minima, \( \theta \) the refractive angle. Here, \( \theta \) is around zero, so it follows that \( \cos \theta \approx 1 \).

\( m_i \) is unknown, but for a maxima,

\[
m_i = m_0 + k_i, \quad k_i = 0, 1, 2, \ldots
\]

Assume \( \nu_i = 1/\lambda_i \), \( \nu_{i+1} = 1/\lambda_{i+1} \), \( \Delta \nu_i = \nu_{i+1} - \nu_i \), and Eq.1 can also be written as

\[
2nd \nu_i = m_0 + k_i
\]
\[2nd \Delta v_i = k_{i+1} - k_i = 1 \quad (4)\]

From Eq. 4, when the effective refractive index \( n \) and the wave number difference \( \Delta v_i \) between the two adjacent extrema from the reflection interference spectrum are known, the actual thickness of PA film could be acquired.

2.2. Determination of the effective refractive index by AFM image

AFM is applied in the system to scan the surface of PA film in order to obtain the porosity. The schematic of the AFM system is shown in Figure 3. The AFM has a resolution of about 1nm. The AFM system consists of probe (optical beam deflection method equipment①, scanner②), pre-amplifier③, control assembly (PID feedback circuit④, scan control circuit⑤, low-voltage and high-voltage amplifier etc.), A/D and D/A interface⑥, computer and software⑦, rough move and micro move mechanism⑧ etc.

After the porosity is determined, the efficient refractive index could be calculated by Maxwell-Garnett effective dielectric constant theory. The porous alumina film has unique structure, which is well-arranged hexagonal cell arrays, and there is a nano-pore in the center of every cell. These pores with uniform size are parallel with each other and vertical with the substrate. The porosity of a hexagonal structure is given by

\[f = \frac{\pi}{2\sqrt{3}} \left( \frac{D_p}{D_{\text{int}}} \right)^2 \quad (5)\]

where \( D_p \) is the pore diameter, \( D_{\text{int}} \) the interpore distance [5].

Then calculate the efficient refractive index by Maxwell-Garnett effective dielectric constant theory,

\[\frac{\varepsilon_{\text{eff}} - \varepsilon_{Alox}}{\varepsilon_{\text{eff}} + 2\varepsilon_{Alox}} = f \frac{\varepsilon_{\text{air}} - \varepsilon_{Alox}}{\varepsilon_{\text{air}} + 2\varepsilon_{Alox}} \quad (6)\]

where \( \varepsilon_{\text{air}} \) and \( \varepsilon_{Alox} \) are the dielectric constant of pure air and pure alumina, \( \varepsilon_{\text{eff}} \) the effective dielectric constant of porous alumina, \( f \) the porosity. It is known to us that \( n_{Alox} \approx 1.65, \varepsilon_{Alox} = n_{Alox}^2 \approx 2.723, \varepsilon_{\text{air}} \approx 1 \).
So we can determine the efficient refractive index based on Eq. 5 and Eq. 6.

3. Experiment and result

Experiment was carried out to determine the feasibility and stability of the system. In the Experiment, we measured a piece of PA sample. The experiment was carried out at atmosphere and ambient temperature.

As for the AFM, a 10mW laser diode was used as the light source, wavelength $\lambda=680$nm, contact mode. The photoelectric detector is S1743 series 2D PSD, and cantilever is “V” shape. As shown is Figure 4, we got the AFM image of the PA film sample in this condition. From the image, we obtained the pore diameter and the interpore distance, $D_p=97$nm, $D_{int}=265$nm. So the porosity can be calculated by Eq. 5, $f=12.1\%$, and the efficient refractive index by Eq. 6, $n_{eff}=1.57$.

In the experiment, we acquired reflective spectrum of two regions of the PA film sample to determine the repeatability of this method. The reflective interference spectrums of two regions are shown in Fig. 5(a), (b).

![AFM image of PA](400 nm)

**Figure 4.** AFM image of PA.

![Reflective interference spectrums](400 nm)

(a) Reflective interference spectrums of the 1st region  (b) Reflective interference spectrums of the 2nd region

**Figure 5.** Reflective interference spectrums of the PA film sample.

Table 1 lists the wavelength of adjacent extrema from the reflective interference spectrum, from which we can calculate the wave number difference $\Delta \nu$ between the two adjacent extrema by $\Delta \nu = \nu_{i+1} - \nu_i$.

| Region | $\lambda_1$ | $\lambda_2$ | $\lambda_3$ | $\lambda_4$ | $\lambda_5$ | $\lambda_6$ | $\lambda_7$ | $\lambda_8$ | $\lambda_9$ | $\lambda_{10}$ |
|--------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|
| 1      | 672         | 658         | 646         | 634         | 622         | 612         | 601         | 590         | 580         | 570         |
| 2      | 672         | 658         | 645         | 633         | 621         | 609         | 598         | 587         | 577         | 567         |

**Table 1.** The wavelength of adjacent extrema from the reflection interference spectrum (nm).
Based on the Averaging-error Effect, the error will decrease when \( \Delta \nu \) is replaced by \( \Delta \nu = (\nu_{10} - \nu_{1}) / 9 \). As for the two regions of PA film sample, from Table 1, we can get \( \Delta \nu \) for each region

\[
\Delta \nu_1 = (1/570 - 1/672)/9 \times 10^6 = 29.59 \text{ mm}^{-1}
\]

\[
\Delta \nu_2 = (1/567 - 1/672)/9 \times 10^6 = 30.62 \text{ mm}^{-1}
\]

As related in the former section, the efficient refractive index \( n_{\text{eff}} = 1.57 \), then by Eq. 4, we can determine the actual thickness of the two regions of PA film sample, \( d_1 = 10.76 \mu \text{m}, d_2 = 10.40 \mu \text{m} \). We find out the relative error is about 3%, which could be accepted. So the system we developed proves good repeatability.

4. Conclusion

We developed a set of thickness measurement for PA film based on AFM and fiber spectrometer, which realizes non-destructive and in-situ measurement with high-resolution. In the experiment, we measured thickness of two regions of PA film sample by the system. The results show that the system has a good repeatability and feasibility.

The system is universal for thickness measurement. In this article, we only presented the measurement for PA film. Actually, the system also fits for measurement for many other films like porous silicon film etc. In the experiment, we only presented the measurement in the air environment, and the pore of PA film is filled with air. The method also can be applied to thickness measurement for porous films in other gas or liquid medium like water or alcohol etc.

References

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