Chapter III – Optical characterizations of thinfilms

Department of Photonics, National Sun Yat-sen University Professor Chu, Ann-kuo The optical measurement includes three different categories:

- Photometric measurements, where amplitude of reflected or transmitted light is measured;
- Interference measurements, where phase of reflected or transmitted light is measured;
- Polarization measurements, where ellipticity of reflected light is measured.

Incident light is reflected, absorbed, emitted, or transmitted.



Optical systems

The Eye



- The lens and cornea remain fixed during the operation of the eyes.
- The focusing is accomplished by bending the lens with eye muscles.
- The brightness of the images is controlled by the iris. Maximum opening of the iris is 7 mm. Optimum opening is 3.5 mm.
- The eye muscles are most relaxed when eye is looking at distant objects (> 5 m). There is a closest point at which the eye can see an object is called the near point. The near point distance is about 25 cm, and is getting longer as a person ages.

Accommodation



Diopter/dioptric power

 $\mathbf{D} = 1/f$ in meter

$$\frac{1}{f} = (n-1)\left(\frac{1}{R_1} - \frac{1}{R_2}\right)$$
$$D = (n-1)\left(\frac{1}{R_1} - \frac{1}{R_2}\right) = D_1 + D_2 = \frac{(n-1)}{R_1} + \frac{-(n-1)}{R_2}$$

Intact unaccommodated eye: f = 58.6 D/1.7 cm Cornea: f = 43 D/2.3 cm Crystalline lens (in air): f = 19 D/5.3 cm

Myopia/negative lenses



Hyperopia/positive lenses



Myopia/negative lenses



Hyperopia/positive lenses

Example: Myopia/negative lens

$$\frac{1}{t_1} = \frac{1}{t_0} + \frac{1}{f_l} \quad t_0 \to \infty$$

$$\frac{1}{t_1} = \frac{1}{f_l} \to t_1 = f_l \quad \therefore t_{01} = d + f_l$$

$$\frac{1}{t_{11}} = \frac{1}{x} = \frac{1}{-t_{01}} + \frac{1}{f_e}$$

$$\Rightarrow x(back_focal_point) = \frac{f_e(d + f_l)}{d + (f_l - f_e)}$$

For contact lens,

$$\frac{1}{f} = \frac{1}{f_c} + \frac{1}{f_e} = \frac{1}{x}$$
$$\Rightarrow \frac{1}{f_c} = \frac{-1}{f_l + d} \quad \longleftarrow \quad \text{Independent of eye itself}$$

For $f_l >> \mathbf{d}$, $\Rightarrow f_c \approx f_l \Rightarrow D_c \approx D_l$

Optical microscope

1. Eyepiece

- Eyes \rightarrow eyepiece \rightarrow optical system
- To balance out the aberration

2. Field stop (FS)

• The element limits the size of the object that can be imaged by the system.

3. Objective/Aperture stop (AS)

- The lens system closest to the object.
- It is also the AS of the microscope that determines the amount of light reaching the image.

Eyepiece/ocular

- A visual optical magnifier
- Eyepiece sees the intermediate image of the object formed by the preceding lens system.
- It provides virtual image of the intermediate image located at or near infinity.

 $\mathbf{MP} = \mathbf{d}_0 D_e \qquad \mathbf{d}_0 = 250 \text{ mm}$ $\mathbf{MP} = 250 \text{ mm}/f_e$

• Chromatic aberration, f is a function of index $n(l) \rightarrow f = f(l)$

Resolution

Raleigh criterion suggest that two object can be distinguished when the center maximum of one image coincides of the first minimum of the other. The intensity between the two peaks then decreases to 80% of the peak height. The resolution based on Raleigh criterion is

 $s = \frac{0.61\lambda}{nsin\theta} = \frac{0.61\lambda}{NA}$

where n and θ are the refractive index and the half angle subtended by the lens at the object. The NA usually engraved on the objective mount. It is a number that expresses the resolving power of the lens and the brightness of the image. For high resolution, that is, small s, NA should be made as large as possible. However, high NA corresponds not only to high resolution but also to shallow depth of field and shallow working distance (the distance from focus point of the object plane to the front surface of the objective. The depth of focus D_{focus} (the thickness of image space that is simultaneously in focus) is given by:

$$D_{focus} = \frac{\lambda}{4(NA)^2}$$

 D_{focus} is insufficient for both the top and the bottom surfaces of an IC to be in focus simultaneously at 200× magnification.

The depth of field D_{field} is the thickness of the object space that is simultaneously in focus, and is given by

$$D_{field} = \frac{\sqrt{n^2 - (NA)^2}}{(NA)^2} \lambda$$

Both D_{focus} and D_{field} decrease with increasing NA, and but the resolution increases. So three variables can be adjusted to reduce s or increase the resolution:

- Wavelength reduction
- Increasing the angle θ_0 toward theoretical maximum (~90°)
- Immersion of objectives in a fluid with higher refractive index than air

Magnification M is related to the resolving power of the microscope and the eye. The images must be magnified sufficiently for detail to be visible to the eye. The resolving power is the ability to reveal of an object by means of eye, microscope. And camera.

$$M = \frac{Maximum \, NA \, of \, microscope}{Minimum \, NA \, of \, eye} = \frac{2.0}{0.002} = 1000 \times$$

Example: MP of an optical microscope

Optical absorptions

The reflection coefficient of a material can be readily obtained from Snell's law, as shown in the following

$$\mathbf{R}^{\mathrm{TE}} = \frac{\sqrt{1 - \sin^2 \theta_i} - \sqrt{1 - \frac{\varepsilon_1}{\varepsilon_2} \sin^2 \theta_i} \sqrt{\frac{\varepsilon_2}{\varepsilon_1}}}{\sqrt{1 - \sin^2 \theta_i} + \sqrt{1 - \frac{\varepsilon_1}{\varepsilon_2} \sin^2 \theta_i} \sqrt{\frac{\varepsilon_2}{\varepsilon_1}}} \qquad \underbrace{\mathbf{Light source}}_{\mathbf{Light source}} \mathbf{Light source} \mathbf{Light source}$$

where $\varepsilon = \varepsilon_r + i \varepsilon_i = (n^2 - \kappa^2) + i 2n\kappa$. n and κ are real part and imaginary part of refractive index. θ_i is the incident angle of the incoming light. If one assumes that the decay of light by absorption of the material depends linearly on the absorption coefficient (a) and the intensity itself, then

$$-\frac{dI(x)}{dx} = \alpha I(x) \quad \text{and} \quad I_t = I_i e^{-\alpha d} \quad \text{or} \quad \alpha = -\frac{1}{d} \ln(\frac{I_t}{I_i})$$

If the reflectivity of the sample surface is taken inti account, then the absorption coefficient α can be written as:

$$\alpha = -\frac{1}{d} \ln[\frac{I_t}{(1-R)^2 I_i}]$$

In other words, if α is known, we can calculated the thickness d of the sample from above equation.

Example: Absorption coefficient of bulk semiconductors

• At short wavelengths, Si is very strongly absorbing. $\alpha(\lambda) > 10^5 \text{ cm}^{-1} \text{ when } \lambda < 0.38 \ \mu\text{m}$

At these wavelengths, the photons are sufficiently energetic to excite electrons directly from valence band to conduction band.

- At longer wavelengths, the photon energy becomes too small to allow such direct transition to occur. One or more phonons are required in the absorption process to maintain momentum conservation.
- The absorption coefficient approaches zero as the photon wavelength becomes longer than that corresponding to the Si bandgap.

Ellipsometry

Ellipsometry is used to measure the thickness of thin dielectric films on highly absorbing substrates but can also be used to determine optical constants, such as film thickness and index of refraction, of thin films or substrates.

- 1. The incident polarized lights can be resolved into a component p, parallel to the plane of incidence and a component s, perpendicular to the plane of incidence.
- 2. If absorption = 0, only the amplitude of the reflected wave is affected.
- 3. If absorption ≠ 0, the reflected wave will generally be reduced in amplitude and shifted in phase → elliptically polarized lights. Complex number

$$r_{p} = \frac{E_{pr}}{E_{pi}} \quad and \quad r_{s} = \frac{E_{sr}}{E_{si}} \qquad \Longrightarrow \qquad \Gamma = \frac{r_{p}}{r_{s}} = \tan(\Psi) \times e^{j\Delta} \qquad \stackrel{0^{\circ} \le \Psi \le 90^{\circ}}{0^{\circ} \le \Delta \le 360^{\circ}}$$

Null Ellipsometry/Substrate

The complex index of refraction of the sample can be determined if n_0 is known and if ellipsometric ratio Γ is measured at the incident angle θ . Note that for a air-thin film-substrate system, the equation becomes much more complicate because they are dependent on n, t, ϕ , and λ .

Null Ellipsometry/Thin Film

- The major application of ellipsometry is for measurements of thickness and index of thin, non-absorbing films on semiconductor substrates.
- In principle, there is no limitation to the thickness of the layer that can be determined. However, uniformity optical properties and a sharp planar film/substrate interface are required.
- The calculations of ellipsometry are based on the Maxwell's equations which generally do not apply to layers only a few atomic layer thick.
- 2π ambiguity of thick layers can cause problems since Ψ and Δ are cyclic function of thickness.

$$t = \frac{\lambda}{2\sqrt{n_1^2 - \sin^2(\phi)}}$$

Full cycle thickness of SiO₂ with $n_1 = 1.465$ at $\lambda = 632.8$ nm and $\phi = 70$ ° is 281.5 nm. For a 50 nm thick SiO₂ film, the same Ψ and Δ will be measured for films of (50+281.5) nm, (50+563) nm, ect.

$\Psi - \Delta$ plots of Ellipsometry

- Each point in the (Ψ,Δ) plane corresponds to a particular pair of values for the film index n₁, and film thickness t.
- The resolution of index measurement is 0.0001.

Transparent films on Si substrate (4.05-j0.028) at λ = 546.1 nm and ϕ = 70 °

$$t = \frac{m\lambda}{2\sqrt{n_1^2 - \sin^2(\phi)}}$$

Example: HR/AR coating for diode lasers

Example: Ta₂O₅ and SiO_x by reactive sputtering

- Refractive index Vavelength
- Oxygen partial pressure Extinction coefficient
- The extinction coefficient is the imaginary part of refractive index.

 $n = n_r + \kappa$ κ Material optical loss

Transmission

Fabry-Perot etalon

k is the wave number of the wave propagating inside the medium. If the medium does not contribute to the field intensity, then the medium is called the passive medium, and k is a real number. If the wave obtains gain (or suffers loss) in side the medium, then the medium is called active medium, and k is a complex number.

$$k = k_0 + j \frac{g - \alpha}{2}$$
 where g and α are gain and loss in cm⁻¹

$$\bullet \left| \mathbf{r}_{1,2} \right|^2 = \mathbf{R}_{1,2}$$

• The absorption coefficient α is related to the complex refractive index $n_1 + j\kappa_1$ by

•
$$T = \frac{4\pi\kappa_1}{\lambda}$$
 κ_1 : extinction coefficient
• $T = \frac{I_t}{I_i} = \frac{(1-R)^2 e^{-\alpha d}}{1+R^2 e^{-2\alpha d} - 2\operatorname{Re}^{-\alpha d}\cos(\phi)}$ where $R = \frac{(n_0 - n_1)^2 + \kappa_1^2}{(n_0 + n_1)^2 + \kappa_1^2}$ and $\phi = \frac{4\pi n_1 d}{\lambda} = \frac{2\pi/\lambda}{\frac{1}{2n_1 d}} = \frac{f}{f_1}$

- If the resolution of the apparatus is sufficiently high, $\Delta f \leq f_1$, the oscillatory transmittance curve is observed. For example, a Si Fabry-Perot etalon with a thickness of 300 µm and index n₁ of 3.42 $\rightarrow \Delta f \leq$ 4.9 cm⁻¹.

$$T = \frac{1}{2\pi} \int_{-\pi}^{\pi} \frac{(1-R)^2 e^{-\alpha d}}{1+R^2 e^{-2\alpha d} - 2\operatorname{Re}^{-\alpha d} \cos(\phi)} d\phi \qquad \Rightarrow T = \frac{(1-R)^2}{1-R^2} = \frac{1-R}{1+R}$$

If $n_{Si} = 3.42$, T = 0.54, and R = 0.3.

• If α and n₁ are constant over the wavelength interval

$$\Rightarrow T = \frac{(1-R)^2 e^{-\alpha d}}{1-R^2 e^{-2\alpha d}} \quad and \quad R = \frac{(n_o - n_1)^2 + \kappa_1^2}{(n_o + n_1)^2 + \kappa_1^2}$$

• Use $\alpha = 4\pi \kappa_1 / \lambda$, the cos ϕ has maxima when $m\lambda_0 = 2n_1 d$

$$\Rightarrow d = \frac{m\lambda_o}{2n_1} = \frac{(m+1)\lambda_1}{2n_1} = \frac{(m+i)\lambda_i}{2n_1} \to m = \frac{i\lambda_i}{\lambda_0 - \lambda_i}$$
$$\Rightarrow d_{m=1} = \frac{1}{2n_1(\frac{1}{\lambda_1} - \frac{1}{\lambda_0})} = \frac{1}{2n_1\Delta(\frac{1}{\lambda_1})}$$

where 1/l is the wave number and $\Delta(1/\lambda)$ is the wave number interval between two maxima or minima of the transmittance curve.

Use
$$n_1 = 3.5$$
, and $\Delta(1/\lambda) = 1.51 \text{ cm}^{-1}$, $d = \frac{1}{2n_1\Delta(1/\lambda)} = 946 \mu m$

• Certain impurities contained in a semiconductor sample exhibit absorption. The transmittance with absorption but no "cos" oscillation is given by

$$T = \frac{(1-R)^2 e^{-\alpha d}}{1-R^2 e^{-2\alpha d}} \quad \Longrightarrow \quad \alpha = -\frac{1}{d} \ln\left[\frac{\sqrt{(1-R)^4 + 4T^2 R^2} - (1-R)^2}{2TR^2}\right]$$

- Absorption due to lattice vibrations, impurities, free-carrier absorption must be considered in the analysis.
- Due to surface roughness, the transmittance becomes wavelength dependent, and T can vary from wafer to wafer. For example, the transmission of wafers with rough surfaces can be written as

 $T \sim e^{-C/\lambda^2} e^{-\alpha d}$

where C characterizes the scattering of rough surface.

Instrument for Transmission Measurement

• Fellget advantage

In monochromator transmission measurement only a small fraction of the entire spectrum is observed at a given time, whereas in FTIR the entire spectrum is observed over the measurement period.

• Jacquinot advantage

In monochromator transmission measurement the among of light passing through the system is limited by the entrance and exit slits, whereas in FTIR relatively large circular apertures are used.

Fourier Transform Infrared Spectroscopy (FTIR)

• The basic optical component of FTIR is the Michelson interferometer. The detector output consists of a series of maximum and minimum that can be described by the equation

• When the source emits more than one wavelength the interferogram, I(x) is replaced by integration

$$I(x) = \int_{0}^{f_{1}} B(f)[1 + \cos(2\pi xf)]df$$

$$B(f): \text{ Source intensity} \quad Af_{1} \quad I(x) = Af_{1} \frac{\sin(2\pi xf_{1})}{2\pi xf_{1}}$$

$$0 \quad f_{1} \quad f \quad Wings \quad Centerbust$$

• The measured quantity in FTIR is the interferogram. It contains not only the spectral information of the source, but also the transmittance characteristics of the sample. But it is the spectral response that is of interest, which can be calculated from the interferogram using the Fourier transform

$$B(f) = \int_{-\infty}^{+\infty} I(x) \cos(2\pi x f) dx$$

Note that B(f) contains the spectral content of the source, the sample, and the ambient in the path of the measurement. It is common practice to reduce atmosphere water and carbon dioxide absorption by purging the apparatus by nitrogen. The effect of the source is eliminated by making one measurement without the sample, background measurement, and one with the sample. The real effect of the sample can be obtained by subtracting the two.

FTIR system

Example: Impurity concentration

- FTIR can be used to detect certain impurities. The most important examples are oxygen and carbon in Si. Interstitial oxygen in Si causes absorption at $\lambda = 9.05 \ \mu m \ (1105 \ cm^{-1})$ at 300 K and 8.87 $\mu m \ (1227.6 \ cm^{-1})$ at 77 K due to vibration of SiO₂ complex.
- The optical absorption coefficient are converted to concentration, N, by

 $\mathbf{N} = \mathbf{C}\boldsymbol{\alpha} \quad (\mathbf{ppma})$

C = 4.9 for oxygen in Si (300K), and C = 0.9 for carbon in Si (77K)

Example: chemical bonding of Tin oxide

FTIR measurement

Example: Film thickness

• A maximum in the interferogram is observed when both optical paths from the BS to the mirrors are identical. For thickness measurement for a layer on a substrate, a second maximum is observed when the movable mirror has moved by a distance equal to the optical path of the layer.

Example: Opaque film thickness

- The simplest method of measuring the thickness of nontransmitting films is by multi-beam interferometry using Fizeau fringes.
- The light incident angle is almost 0 radians, so that all the reflected waves almost coincide spatially.
- Assuming that there is a phase change π at each reflection, the change in phase during a path travel is

$$\psi = \frac{4T\pi}{\lambda} + 2\pi = 2N\pi$$
 or $T = \frac{(N-1)\lambda}{2}$

- The condition for constructive interference is that the phase change be an integer of 2π and the distance between maximum of successive fringes corresponds to a distance $T = \lambda/2$.
- A small angle between the film-coated substrate and the reflected plate translates the changes in the vertical dimension to horizontal displacement of the fringes.

- The fringes spacing is essentially $\lambda/2$ when the incident illumination is almost normal to the surface and the wedge angle between the film and reflector is small.
- The film thickness, t, is measured by introducing a step into the film which is then coating a thin layer of highly reflected material. This step abruptly changes the distance T and causes a abrupt fringe displacement. The thickness t is measured by comparing the fringe displacement to the fringe spacing. So

$$t = \frac{d}{D} \cdot \frac{\lambda}{2}$$

where D is the fringe separation and d is the fringe displacement due to the step.

• The resolution of this method is about 1/400 because for highly reflecting surfaces the width of a fringe is about D/40, and the displacements of about one-fifth of a fringe width can be measured,

Reflection

- Reflectivity measurement is common used to determined layer thickness, both for insulating layers on semiconducting substrates and for epitaxial semiconductor films.
- The reflectance of a non-absorbing layer on a non-absorbing substrate is

$$R = \frac{r_1^2 + r_2^2 + 2r_1r_2\cos(\phi_1)}{1 + r_1^2r_2^2 + 2r_1r_2\cos(\phi_1)}$$

$$r_1 = \frac{n_o - n_1}{n_o + n_1} \quad and \quad r_2 = \frac{n_1 - n_2}{n_1 + n_2}$$

$$\phi_1 = \frac{4\pi n_1 t_1\cos(\phi')}{\lambda} \quad and \quad \phi' = \sin^{-1}[\frac{n_o\sin(\phi)}{n_1}]$$

• The reflectance exhibits maximum at wavelengths

$$\lambda_{\max} = \frac{2n_1 t_1 \cos(\phi')}{m} \qquad m = 1, 2, 3...$$
$$t_1 = \frac{i\lambda_o \lambda_i}{2n_1(\lambda_i - \lambda_o)\cos(\phi')} \qquad \text{where if peaks the set of th$$

where i is the number of complete cycles from λ_0 to λ_i , the two wavelengths peaks that bracket the i cycles.

• For i = 1, from the figure

 $\lambda_{o} = 0.32 \ \mu m \text{ and } \lambda_{1} = 0.384 \ \mu m \longrightarrow t_{1} = 0.75 \ \mu m$

- Instead of illuminating the sample with monochromatic light and changing wavelength, it is possible to shine white light, containing many wavelengths, onto the sample and analyze the reflected light by passing it through a spectrometer.
- Reflectance measurements are also used to determine the thickness of epitaxial semiconductor layers. But the technique only works if there is a substantial doping change at the layer-substrate interface because there must be a measurable index change at the interface.
- The quantity 2n₁cos(\$\phi'\$) is a constant determined by experiment set up and the index of the film

$$2n_1\cos(\phi') = 2\sqrt{n_1^2 - n_o^2\sin^2(\phi)}$$

• For thinner films (< 200 nm) it is difficult to find the first minimum unless extremely short wavelengths are used.

Prism Coupler

The prism coupler, known from experiments on integrated optics, can be used to determine the refractive index and the thickness of a light-guiding thin film. Both parameters are obtained simultaneously and with good accuracy by measuring the coupling angles at the prism and fitting them by a theoretical dispersion curve.

- **BK7**, and TiO₂ are the common materials of the prism.
- Prism coupler is suitable for measurement of relatively thick dielectric films.
- Optically flat surface of the sample is required.
- The resolution of index measurement is 0.0005.

Slab waveguide

We will concentrate on analyzing 1-D guiding medium, that is, the refractive index only varies in one direction.

For a electromagnetic disturbance to be a mode, it must retain its shape when being propagated forward. That is, the transverse dependence of the field does not change. A translation along propagation direction results in a phase shift.

$$\implies \vec{E}(x, y, z, t) = \operatorname{Re}[\vec{E}(x, y)e^{-i\omega t}e^{i\beta z}] \rightarrow \frac{\partial \vec{E}}{\partial z} = i\beta \vec{E}$$

Geometric optics of slab waveguide

Assuming $\vec{E}(x,z) = E_0 e^{ik_x x} e^{ik_z z}$ So the total phase shift: $\Delta \phi_{A-B} = k_x 2d + k_z \Delta z + \delta_{12} + \delta_{23}$

Propagation of high order modes in a waveguide

Example: Numerical aperture (NA)

Snell law: $n_0 \sin \theta_0 = n_2 \sin \theta_2$, and $\theta_i = \pi/2 - \theta_2$ For minimum loss alone z direction,

$$\theta_i > \theta_c = \sin^{-1} \frac{n_1}{n_2} \Big|_{cut-off}$$

$$NA = \sin \theta_0 \Big|_{cutoff} = \frac{n_2}{n_o} \sin \theta_2 = \frac{n_2}{n_o} \cos \theta_c = \frac{n_2}{n_o} \sqrt{1 - \frac{n_1^2}{n_2^2}} = \frac{\sqrt{n_2^2 - n_1^2}}{n_0}$$

Measurement of prism coupler

Bevel - Stain technique

Groove and stain technique

$$rightarrow t = N \lambda/2$$

• The thickness determination is independent of the bevel angle since interference fringes occur every $\lambda/2$.

 $\implies t = \sqrt{R^2 - (\frac{W_2}{2})^2} - \sqrt{R^2 - (\frac{W_1}{2})^2}$

t = W²/8R Valid for (W/2)² << R² **Index-to-temperature Coefficient (dn/dT) Measurement**

Measurement Results

II. SiO₂/Ta₂O₅/SiO₂ waveguide

I. Ta₂O₅/SiO₂/Ta₂O₅ waveguide